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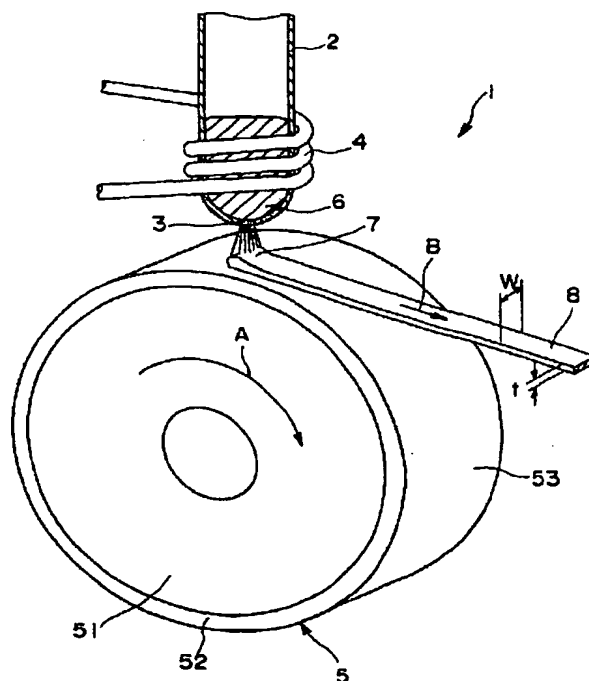
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(54) 【発明の名称】 磁石材料の製造方法、磁石材料およびボンド磁石

(57) 【要約】

【課題】 高い磁気特性が得られる磁石材料の製造方法、磁石材料およびボンド磁石を提供すること。

【解決手段】 急冷薄帯製造装置1は、筒体2と、加熱用のコイル4と、筒体2に対し回転する冷却ロール5とを備えている。筒体2の下端には、磁石材料の溶湯6を射出するノズル3が形成されている。雰囲気ガス中で、溶湯6をノズル3から射出し、回転する冷却ロール5の周面53に衝突させ、冷却固化して、急冷薄帯8を製造する。この場合、冷却ロール5の周面53を構成する金属材料は、該金属材料の水平表面上に溶湯6の液滴を置き、凝固させたとき、その凝固物の前記水平表面とのなす接触角が70°～170°となるような濡れ性を有している。



## 【特許請求の範囲】

【請求項 1】 磁石材料の溶湯をノズルから射出し、前記ノズルに対し回転している冷却ロールの周面に衝突させ、冷却固化して、薄帯状の磁石材料を製造する磁石材料の製造方法であって、

前記冷却ロールの周面を構成する金属材料は、該金属材料の水平表面上に前記溶湯の液滴を置き、凝固させたとき、その凝固物の前記水平表面とのなす接触角が  $70 \sim 170^\circ$  となるものであることを特徴とする磁石材料の製造方法。

【請求項 2】 雰囲気ガス中で磁石材料の溶湯をノズルから射出し、前記ノズルに対し回転している冷却ロールの周面に衝突させ、冷却固化して、薄帯状の磁石材料を製造する磁石材料の製造方法であって、前記冷却ロールの周面を構成する金属材料は、前記雰囲気ガスと同種および同圧の気体中で前記金属材料の水平表面上に前記溶湯の液滴を置き、凝固させたとき、その凝固物の前記水平表面とのなす接触角が  $70 \sim 170^\circ$  となるものであることを特徴とする磁石材料の製造方法。

【請求項 3】 前記冷却ロールの周速度が、 $1 \sim 60 \text{ m/秒}$  である請求項 1 または 2 に記載の磁石材料の製造方法。

【請求項 4】 前記冷却ロールの回転に伴う冷却ロール周面の最大偏心量が、得られる薄帯状の磁石材料の平均厚さの 2 倍以下である請求項 1 ないし 3 のいずれかに記載の磁石材料の製造方法。

【請求項 5】 前記雰囲気ガスは、不活性ガスである請求項 1 ないし 4 のいずれかに記載の磁石材料の製造方法。

【請求項 6】 前記磁石材料は、R（ただし、R は、Y を含む希土類元素のうちの少なくとも 1 種）を含む合金である請求項 1 ないし 5 のいずれかに記載の磁石材料の製造方法。

【請求項 7】 前記磁石材料は、R（ただし、R は、Y を含む希土類元素のうちの少なくとも 1 種）と TM（ただし、TM は、遷移金属のうちの少なくとも 1 種）と B を含む合金である請求項 1 ないし 5 のいずれかに記載の磁石材料の製造方法。

【請求項 8】 請求項 1 ないし 7 のいずれかに記載の磁石材料の製造方法により製造されたことを特徴とする薄帯状の磁石材料。

【請求項 9】 請求項 8 に記載の磁石材料を粉砕して粉末状としたことを特徴とする粉末状の磁石材料。

【請求項 10】 請求項 9 に記載の粉末状の磁石材料を結合樹脂で結合してなることを特徴とするボンド磁石。

【請求項 11】 前記粉末状の磁石材料の含有量が  $82 \sim 99.5 \text{ wt\%}$  である請求項 10 に記載のボンド磁石。

【請求項 12】 保磁力  $iH_c$  が  $0.35 \text{ MA/m}$  以上である請求項 10 または 11 に記載のボンド磁石。

【請求項 13】 磁気エネルギー積  $(BH)_{\text{max}}$  が  $50 \text{ kJ/m}^3$  以上である請求項 10 ないし 12 のいずれかに記載のボンド磁石。

## 【発明の詳細な説明】

## 【0001】

【発明の属する技術分野】本発明は、磁石材料の製造方法、磁石材料およびボンド磁石に関するものである。

## 【0002】

【従来の技術】磁石材料として、希土類元素を含む合金で構成される希土類磁石材料は、高い磁気特性を有するため、モータ等に用いられた場合に、高性能を発揮する。

【0003】このような磁石材料は、例えば急冷薄帯製造装置を用いた急冷法により製造される。この製造方法は、次の通りである。

【0004】所定の合金組成の磁石材料（以下「合金」と言う）を溶融し、その溶湯をノズルから射出し、ノズルに対して回転している冷却ロールの周面に衝突させ、該周面と接触させることにより合金を急冷、凝固し、薄帯状（リボン状）の合金を連続的に形成する。この薄帯状の合金は、急冷薄帯と呼ばれる。

【0005】ところで、ノズルから射出された溶湯は、冷却ロールの周面に衝突して、まずパドル（湯溜り）を形成し、その後冷却されて凝固するが、その冷却速度が遅いと、結晶粒が粗大化し、磁気特性が低下する。

【0006】そのため、冷却ロールの周面を構成する金属材料としては、熱伝導性に優れる材料が選択されていた。

## 【0007】

【発明が解決しようとする課題】しかしながら、近年、冷却ロールの周面に熱伝導性に優れた金属材料を用いた場合でも、磁気特性が低い場合があるという問題が生じている。

【0008】本発明の目的は、高い磁気特性が得られる磁石材料の製造方法、磁石材料およびボンド磁石を提供することにある。

## 【0009】

【課題を解決するための手段】急冷薄帯の体積流量  $Q$ （単位時間当たりに製造される急冷薄帯の体積＝単位時間当たりに射出される溶湯の体積）は、急冷薄帯の幅  $w$ 、厚さ  $t$ 、冷却ロールの周速度  $V$  としたとき、下記式（1）で表わされる。

$$【0010】 Q = w \times t \times V \quad \dots (1)$$

一方、冷却ロールの周面の溶湯に対する濡れ性（以下単に「ロール周面の濡れ性」と言う）が良好であると、パドルが冷却ロールの周面上でより広い面積に広がろうとするので、急冷薄帯の幅  $w$  は大きくなり、逆に、ロール周面の濡れ性が悪いと、急冷薄帯の幅  $w$  は小さくなる。

【0011】従って、冷却ロールの周速度  $V$  および急冷薄帯の体積流量  $Q$  を一定として急冷薄帯を製造した場

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合、上記式(1)から、ロール周囲の濡れ性が良いと、幅 $w$ が大きく、厚さ $t$ が小さい急冷薄帯が得られ、逆にロール周囲の濡れ性が悪いと、幅 $w$ が小さく、厚さ $t$ が大きい急冷薄帯が得られこととなる。

【0012】そして、急冷薄帯の厚さ $t$ が小さいと、厚さ方向の熱伝達が短時間でなされ、結晶粒の微細化にとって有利であるが、急冷薄帯の厚さ $t$ が大きいと、厚さ方向の熱伝達性が悪く、特に急冷薄帯のロール面(冷却ロールの周囲と接触する側の面)とフリー面(冷却ロールの周囲と接触しない側の面)との冷却速度の差が大きくなり、フリー面側において結晶粒が粗大化し易くなる。

【0013】このようなことから、本発明者は、ロール周囲の濡れ性に着目し、鋭意研究を行った結果、ロール周囲の濡れ性を所定の範囲とした冷却ロールを用いることにより、結晶粒の微細化が図れ、優れた磁気特性が得られることを見出し、本発明に至った。

【0014】すなわち、本発明は、下記(1)～(13)に示す通りである。

【0015】(1) 磁石材料の溶湯をノズルから射出し、前記ノズルに対し回転している冷却ロールの周囲に衝突させ、冷却固化して、薄帯状の磁石材料を製造する磁石材料の製造方法であって、前記冷却ロールの周囲を構成する金属材料は、該金属材料の水平表面上に前記溶湯の液滴を置き、凝固させたとき、その凝固物の前記水平表面とのなす接触角が $70 \sim 170^\circ$ となるものであることを特徴とする磁石材料の製造方法。

【0016】(2) 雰囲気ガス中で磁石材料の溶湯をノズルから射出し、前記ノズルに対し回転している冷却ロールの周囲に衝突させ、冷却固化して、薄帯状の磁石材料を製造する磁石材料の製造方法であって、前記冷却ロールの周囲を構成する金属材料は、前記雰囲気ガスと同種および同圧の気体中で前記金属材料の水平表面上に前記溶湯の液滴を置き、凝固させたとき、その凝固物の前記水平表面とのなす接触角が $70 \sim 170^\circ$ となるものであることを特徴とする磁石材料の製造方法。

【0017】(3) 前記冷却ロールの周速度が、 $1 \sim 60 \text{ m/秒}$ である上記(1)または(2)に記載の磁石材料の製造方法。

【0018】(4) 前記冷却ロールの回転に伴う冷却ロール周囲の最大偏心量が、得られる薄帯状の磁石材料の平均厚さの2倍以下である上記(1)ないし(3)のいずれかに記載の磁石材料の製造方法。

【0019】(5) 前記雰囲気ガスは、不活性ガスである上記(1)ないし(4)のいずれかに記載の磁石材料の製造方法。

【0020】(6) 前記磁石材料は、R(ただし、Rは、Yを含む希土類元素のうちの少なくとも1種)を含む合金である上記(1)ないし(5)のいずれかに記載の磁石材料の製造方法。

【0021】(7) 前記磁石材料は、R(ただし、Rは、Yを含む希土類元素のうちの少なくとも1種)とTM(ただし、TMは、遷移金属のうちの少なくとも1種)とBを含む合金である上記(1)ないし(5)のいずれかに記載の磁石材料の製造方法。

【0022】(8) 上記(1)ないし(7)のいずれかに記載の磁石材料の製造方法により製造されたことを特徴とする薄帯状の磁石材料。

【0023】(9) 上記(8)に記載の磁石材料を粉碎して粉末状としたことを特徴とする粉末状の磁石材料。

【0024】(10) 上記(9)に記載の粉末状の磁石材料を結合樹脂で結合してなることを特徴とするボンド磁石。

【0025】(11) 前記粉末状の磁石材料の含有量が $82 \sim 99.5 \text{ wt\%}$ である上記(10)に記載のボンド磁石。

【0026】(12) 保磁力 $iH_c$ が $0.35 \text{ MA/m}$ 以上である上記(10)または(11)に記載のボンド磁石。

【0027】(13) 磁気エネルギー積 $(BH)_{\text{max}}$ が $50 \text{ kJ/m}^3$ 以上である上記(10)ないし(12)のいずれかに記載のボンド磁石。

【0028】

【発明の実施の形態】以下、本発明の磁石材料の製造方法、磁石材料およびボンド磁石について、添付図面を参照しつつ詳細に説明する。

【0029】図1は、本発明の磁石材料を単ロール法により製造する装置(急冷薄帯製造装置)の構成例を示す斜視図、図2は、図1に示す装置における溶湯の冷却ロールへの衝突部位付近の状態を示す断面側面図である。

【0030】図1に示すように、急冷薄帯製造装置1は、磁石材料を収納し得る筒体2と、該筒体2に対し図中矢印A方向に回転する冷却ロール5とを備えている。筒体2の下端には、磁石材料の溶湯を射出するノズル(オリフィス)3が形成されている。

【0031】また、筒体2のノズル3近傍の外周には、加熱用のコイル4が配置され、このコイル4に例えば高周波を印加することにより、筒体2内を加熱(誘導加熱)し、筒体2内の磁石材料を溶融状態にする。

【0032】冷却ロール5は、基部51と、冷却ロール5の周囲53を形成する表面層52とで構成されている。

【0033】基部51の構成材料は、表面層52と同じ材質で一体構成されていてもよく、また、表面層52とは異なる材質で構成されていてもよい。

【0034】基部51の構成材料は、特に限定されないが、表面層52の熱をより速く放散できるように、例えば銅または銅系合金のような熱伝導率の高い金属材料で構成されているのが好ましい。

【0035】また、表面層52は、以下に述べるような

金属材料で構成されているのが好ましい。

【0036】このような急冷薄帯製造装置1は、チャンパー（図示せず）内に設置され、該チャンパー内に、好ましくは不活性ガスやその他の雰囲気ガスが充填された状態で作動する。特に、急冷薄帯8の酸化を防止するために、雰囲気ガスは、不活性ガスであるのが好ましい。

【0037】不活性ガスとしては、例えばアルゴンガス、ヘリウムガス、窒素ガス等が挙げられるが、特にヘリウムガスが好ましい。その理由は、雰囲気ガスとしてヘリウムガスを用いると、急冷薄帯8のロール面81にガス流の巻き込みによるディンプル、特に面積が $2000\mu\text{m}^2$ 以上の巨大ディンプル13（図2中仮想線で示す）が生じ難くなり、熱伝達性が向上して、より高い磁気特性が得られるからである。

【0038】急冷薄帯製造装置1では、筒体2内に磁石材料を入れ、コイル4により加熱して熔融し、その溶湯6をノズル3から射出すると、図2に示すように、溶湯6は、冷却ロール5の周面53に衝突し、パドル（湯溜り）7を形成した後、回転する冷却ロール5の周面53に引きずられつつ急速に冷却されて凝固し、急冷薄帯8が連続的または断続的に形成される。このようにして形成された急冷薄帯8は、やがて、そのロール面81が周面53から離れ、図1中の矢印B方向に進行する。なお、図2中、溶湯の凝固界面71を点線で示す。

【0039】冷却ロール5の周速度Vは、合金溶湯の組成、周面53の溶湯6に対する濡れ性等によりその好適な範囲が異なるが、通常、 $1\sim60\text{m}/\text{秒}$ であるのが好ましく、 $5\sim40\text{m}/\text{秒}$ であるのがより好ましい。冷却ロール5の周速度が遅すぎると、急冷薄帯8の体積流量Qによっては、急冷薄帯8の厚さtが厚くなり（前記式（I）参照）、結晶粒径が増大し、逆に冷却ロール5の周速度Vが速すぎると、非晶質となり、いずれの場合にも、磁気特性が低下する。

【0040】冷却ロール5の周面53を構成する金属材料、すなわち表面層52を構成する金属材料（以下「ロール周面材料」と言う）は、次のような溶湯6に対する濡れ性（以下単に「濡れ性」と言う）を有するもので構成されている。すなわち、図3に示すように、ロール周面材料9で水平表面10を形成し、該水平表面10上に溶湯6の液滴を置き、凝固させたとき、その凝固物11の水平表面10とのなす接触角 $\theta$ が $70\sim170^\circ$ となるものである。この場合、接触角 $\theta$ は、 $80\sim165^\circ$ であるのが好ましく、 $90\sim160^\circ$ であるのがより好ましく、 $95\sim150^\circ$ であるのがさらに好ましい。

【0041】ここで、冷却ロール5の周面53の濡れ性を直接測定せず、それと同一材料（ロール周面材料）で水平表面10を形成し、該水平表面10の濡れ性を測定するのは、周面53は湾曲凸面であるため、溶湯6の液滴を一定の位置に止めて置くことができず、接触角の測定が不可能または困難だからである。

【0042】なお、接触角 $\theta$ の測定に際しては、急冷薄帯8の実際の製造に使用される冷却ロール5の周面53の濡れ性との対応関係をより正確に得るために、溶湯6の液滴の凝固は、前記雰囲気ガスと同種および同圧の気体中で行うのが好ましい。また、凝固物11の体積は、 $0.005\sim0.1\text{cm}^3$ の範囲で測定するのが好ましい。

【0043】接触角 $\theta$ が上記範囲の上限値を超えると、周面53の濡れ性が悪く、急冷薄帯8の体積流量Qによっては、急冷薄帯8の厚さtが厚くなる傾向となり、特に急冷薄帯8のフリー面82側における結晶粒が粗大化し、磁気特性が低下する。なお、この場合でも、体積流量Qを小さくすれば、厚さtも薄くなり（前記式（I）参照）、かかる欠点は解消または緩和されるが、生産性の低下を招くので、好ましくない。

【0044】また、接触角 $\theta$ が上記範囲の下限値未満であると、周面53の濡れ性が良すぎるため、パドル7が広がりすぎ、そのため、急冷薄帯8の形状、寸法（幅w、厚さt）が不安定となり、均一、均質な急冷薄帯8が得られない（結晶粒の状態や磁気特性等にバラツキが生じる）。

【0045】なお、接触角 $\theta$ の測定に際し、溶湯6の液滴が水平表面10と接触する凝固界面付近では、凝固収縮により、図4に示すような剥離（浮き上がり）12が生じることがある。この場合には、剥離12の生じた部分を除外して接触角 $\theta$ を測定する。すなわち、剥離12の上端（頂点）を通る水平表面10に平行な面10'を基準面として接触角 $\theta$ を測定する。

【0046】ところで、急冷薄帯製造装置1においては、冷却ロール5自体の寸法精度（真円度）や、冷却ロール5の軸受けに対する取り付け精度等から、冷却ロール5が回転するに際し、図5に示すように、若干の偏心（軸振れ）が生じる。

【0047】この偏心が大きいと、パドル7における溶融合金の表面や凝固界面71が振動し、得られた急冷薄帯8の寸法（幅w、厚さt）に変動が生じたり、急冷薄帯8のロール面81が冷却ロール5の周面53と接触している時間に変動が生じたりする。さらに、前記巨大ディンプル13の発生率も高まる。その結果、急冷薄帯8の冷却速度等が変動し、磁気特性にバラツキが生じる。そして、このような急冷薄帯8から得られた磁石粉末やそれを用いたボンド磁石も、磁気特性が低下する。

【0048】このようなことを防止するために、本発明では、冷却ロール5の回転に伴う冷却ロール5の周面53の最大偏心量 $\Delta R$ （図5参照）を、得られる急冷薄帯8の厚さ（平均値）tの2倍以下とするのが好ましく、1.5倍以下とするのがより好ましく、1倍以下とするのがさらに好ましい。これにより、得られた急冷薄帯8の磁気特性をより均一にすることができる。そして、これより製造されたボンド磁石の磁気特性を高めることが

できる。特に、本発明では、このような最大偏心量 $\Delta R$ を規定することと、前述した周面53の濡れ性を規定することとの相乗効果により、さらに優れた磁気特性を発揮することができる。

【0049】ここで、最大偏心量 $\Delta R$ の下限値は、特に限定されないが、冷却ロール5の周面53の加工精度の限界や、冷却ロール5を支持する軸受けの精度の限界から、 $0.1\mu\text{m}$ 程度とすることができる。

【0050】なお、最大偏心量 $\Delta R$ は、例えば、レーザ変位計、静電式変位計、精密ゲージ等の精密寸法測定機器により測定することができる。

【0051】本発明における磁石材料としては、R（ただし、Rは、Yを含む希土類元素のうちの少なくとも1種）を含む合金、特にR（ただし、Rは、Yを含む希土類元素のうちの少なくとも1種）とTM（ただし、TMは、遷移金属のうちの少なくとも1種）とBとを含む合金のような希土類磁石材料が挙げられ、次の[1]～[4]の組成のものが好ましい。

【0052】[1] Smを主とする希土類元素と、Coを主とする遷移金属とを基本成分とするもの（以下、Sm-Co系合金と言う）。

【0053】[2] R（ただし、RはYを含む希土類元素のうちの少なくとも1種）と、Feを主とする遷移金属と、Bとを基本成分とするもの（以下、R-Fe-B系合金と言う）。

【0054】[3] Smを主とする希土類元素と、Feを主とする遷移金属と、Nを主とする格子間元素とを基本成分とするもの（以下、Sm-Fe-N系合金と言う）。

[4] R（ただし、RはYを含む希土類元素のうちの少なくとも1種）とFe等の遷移金属とを基本成分とし、ナノメートルレベルで磁性相を有するもの（ナノ結晶磁石）。

【0055】Sm-Co系合金の代表的なものとしては、SmCo<sub>5</sub>、Sm<sub>2</sub>TM<sub>17</sub>（ただしTMは、遷移金属）が挙げられる。

【0056】R-Fe-B系合金の代表的なものとしては、Nd-Fe-B系合金、Pr-Fe-B系合金、Nd-Pr-Fe-B系合金、Ce-Nd-Fe-B系合金、Ce-Pr-Nd-Fe-B系合金、これらにおけるFeの一部をCo、Ni等の他の遷移金属で置換したもの等が挙げられる。

【0057】Sm-Fe-N系合金の代表的なものとしては、Sm<sub>2</sub>Fe<sub>17</sub>合金を窒化して作製したSm<sub>2</sub>Fe<sub>17</sub>N<sub>3</sub>が挙げられる。

【0058】前記希土類元素としては、Y、La、Ce、Pr、Nd、Pm、Sm、Eu、Gd、Tb、Dy、Ho、Er、Tm、Yb、Lu、ミッシュメタルが挙げられ、これらを1種または2種以上含むことができる。また、前記遷移金属としては、Fe、Co、Ni等

が挙げられ、これらを1種または2種以上含むことができる。また、磁気特性を向上させるために、磁石材料中には、必要に応じ、B、Al、Cu、Ga、Si、Ti、V、Ta、Zr、Nb、Mo、Hf、Ag、Zn、P、Ge等を含有することもできる。

【0059】以上のような製造方法により得られた本発明の急冷薄帯（薄帯状の磁石材料）8は、結晶粒が微細化され、その結果、優れた磁気特性が得られる。

【0060】また、このような急冷薄帯8を粉砕することにより、本発明の粉末状の磁石材料（磁石粉末）が得られる。

【0061】粉砕の方法は、特に限定されず、例えばボールミル、振動ミル、ジェットミル、ピンミル等の各種粉砕装置、破砕装置を用いて行うことができる。この場合、粉砕は、酸化を防止するために、真空または減圧状態下（例えば $1 \times 10^{-1} \sim 1 \times 10^{-6}$  Torr）、あるいは窒素ガス、アルゴンガス、ヘリウムガス等の不活性ガス中のような、非酸化性雰囲気中で行うこともできる。

【0062】このような磁石粉末は、同一組成のもののみならず、異なる2種以上の組成の磁石粉末を混合したものでもよい。例えば、前記[1]～[4]の組成のものうち、少なくとも2種を混合したものが挙げられる。この場合、混合する各磁石粉末の利点を併有することができ、より優れた磁気特性を容易に得ることができる。

【0063】また、磁石粉末の平均粒径は、特に限定されないが、後述するボンド磁石を製造するためのものの場合、 $0.5 \sim 60\mu\text{m}$ 程度が好ましく、 $1 \sim 40\mu\text{m}$ 程度がより好ましい。また、後述するような少量の結合樹脂で成形時の良好な成形性を得るために、磁石粉末の粒径分布は、ある程度分散されている（バラツキがある）のが好ましい。これにより、得られたボンド磁石の空孔率を低減することができ、ボンド磁石の機械的強度をより高め、磁気特性をさらに向上することができる。

【0064】なお、異なる2種以上の組成の磁石粉末を混合したものの場合、混合する磁石粉末の組成毎に、その平均粒径が異なってもよい。また、このような混合粉末の場合、異なる2種以上の組成の磁石粉末のうちの少なくとも1種が前述した本発明の方法により製造されたものであればよい。

【0065】以上のような磁石粉末を用いてボンド磁石を製造した場合、そのような磁石粉末は、結合樹脂との結合性（結合樹脂の濡れ性）が良く、そのため、このボンド磁石は、機械的強度が高く、熱安定性（耐熱性）、耐食性が優れたものとなる。従って、当該磁石粉末は、ボンド磁石の製造に適している。

【0066】なお、本発明の磁石粉末（粉末状の磁石材料）は、ボンド磁石の製造に用いるものに限定されず、例えば、焼結磁石の製造に用いるものであってもよいこ

とは、言うまでもない。

【0067】次に、本発明のボンド磁石について説明する。

【0068】本発明のボンド磁石は、前述の磁石粉末を結合樹脂で結合してなるものである。

【0069】結合樹脂（バインダー）としては、熱可塑性樹脂、熱硬化性樹脂のいずれでもよい。

【0070】熱可塑性樹脂としては、例えば、ポリアミド（例：ナイロン6、ナイロン46、ナイロン66、ナイロン610、ナイロン612、ナイロン11、ナイロン12、ナイロン6-12、ナイロン6-66）、熱可塑性ポリイミド、芳香族ポリエステル等の液晶ポリマー、ポリフェニレンオキシド、ポリフェニレンサルファイド、ポリエチレン、ポリプロピレン、エチレン-酢酸ビニル共重合体等のポリオレフィン、変性ポリオレフィン、ポリカーボネート、ポリメチルメタクリレート、ポリエチレンテレフタレート、ポリブチレンテレフタレート等のポリエステル、ポリエーテル、ポリエーテルエーテルケトン、ポリエーテルイミド、ポリアセタール等、またはこれらを主とする共重合体、ブレンド体、ポリマーアロイ等が挙げられ、これらのうちの1種または2種以上を混合して用いることができる。

【0071】これらのうちでも、成形性が特に優れており、機械的強度が高いことから、ポリアミド、耐熱性向上の点から、液晶ポリマー、ポリフェニレンサルファイドを主とするものが好ましい。また、これらの熱可塑性樹脂は、磁石粉末との混練性にも優れている。

【0072】このような熱可塑性樹脂は、その種類、共重合化等により、例えば成形性を重視したものや、耐熱性、機械的強度を重視したものというように、広範囲の選択が可能となるという利点がある。

【0073】一方、熱硬化性樹脂としては、例えば、ビスフェノール型、ノボラック型、ナフタレン系等の各種エポキシ樹脂、フェノール樹脂、ユリア樹脂、メラミン樹脂、ポリエステル（不飽和ポリエステル）樹脂、ポリイミド樹脂、シリコン樹脂、ポリウレタン樹脂等が挙げられ、これらのうちの1種または2種以上を混合して用いることができる。

【0074】これらのうちでも、成形性が特に優れており、機械的強度が高く、耐熱性に優れるという点から、エポキシ樹脂、フェノール樹脂、ポリイミド樹脂、シリコン樹脂が好ましく、エポキシ樹脂が特に好ましい。また、これらの熱硬化性樹脂は、磁石粉末との混練性、混練の均一性にも優れている。

【0075】なお、使用される熱硬化性樹脂（未硬化）は、室温で液状のものでも、固形（粉末状）のものでもよい。

【0076】このような本発明のボンド磁石は、例えば次のようにして製造される。磁石粉末と、結合樹脂と、必要に応じ添加剤（酸化防止剤、潤滑剤等）を含むボ

ンド磁石用組成物（コンパウンド）を製造し、このボンド磁石用組成物を用いて、圧縮成形、押出成形、射出成形等の方法により、磁場中または無磁場中で所望の磁石形状に成形する。結合樹脂が熱硬化性樹脂の場合には、成形後、加熱等によりそれを硬化する。

【0077】ボンド磁石中の磁石粉末の含有量は、82～99.5wt%程度であるのが好ましく、90～99wt%程度であるのがより好ましい。特に、ボンド磁石が圧縮成形により製造されたものの場合には、磁石粉末の含有量は、93～99.5wt%程度であるのが好ましく、95～99wt%程度であるのがより好ましい。

【0078】磁石粉末の含有量が少なすぎると、磁気特性（特に磁気エネルギー積）の向上が図れず、また、磁石粉末の含有量が多すぎると、相対的に結合樹脂の含有量が少なくなり、成形性が低下する。

【0079】このような本発明のボンド磁石は、その原材料となる前述した急冷薄帯8の特性や、ボンド磁石の製造条件、ボンド磁石に含まれる磁石粉末の含有量の多さ等から、優れた磁気特性を発揮する。

【0080】すなわち、本発明のボンド磁石は、保磁力  $iH_c$  が好ましくは0.35MA/m以上、より好ましくは0.50MA/m以上である。

【0081】本発明のボンド磁石、特に無磁場中で成形されたボンド磁石は、磁気エネルギー積(BH)<sub>max</sub> が好ましくは50kJ/m<sup>3</sup>以上、より好ましくは70kJ/m<sup>3</sup>以上である。

【0082】本発明のボンド磁石の形状、寸法等は特に限定されず、例えば、形状に関しては、例えば、円柱状、角柱状、円筒状（リング状）、円弧状、平板状、湾曲板状等のあらゆる形状のものが可能であり、その大きさも、大型のものから超小型のものまであらゆる大きさのものが可能である。

【0083】

【実施例】以下、本発明の具体的実施例について説明する。

【0084】（実施例1）合金組成がNd<sub>10</sub>Pr<sub>2.5</sub>Fe<sub>61.1</sub>Co<sub>6</sub>Al<sub>3</sub>Cu<sub>1.5</sub>Nb<sub>1</sub>Ga<sub>1</sub>B<sub>5</sub>（組成A）で表わされる母合金インゴットを鋳造した。このインゴットから約15gのサンプルを切り出した。

【0085】図1に示す構成の急冷薄帯製造装置1を用意し、底部にノズル（円孔オリフィス）を設けた石英管内に前記サンプルを入れた。急冷薄帯製造装置1が収納されているチャンバー内を脱気した後、雰囲気ガスとしてヘリウムガスを導入し、温度21℃、圧力60KPaの雰囲気ガスとした。

【0086】その後、石英管内のインゴットサンプルを高周波誘導加熱により熔融し、この溶湯を、1500rpm（周速度：15.7m/秒）で回転する直径200mm、幅20mmの冷却ロールの周面に向けて、石英管の内

圧と雰囲気圧との差圧により噴射し、前記組成Aの合金の急冷薄帯を得た。

【0087】冷却ロールの表面層（ロール周面）は、Pd-8wt%Ru-2wt%Pt合金で構成されたものとした。また、この表面層の厚さは、5mmとした。

【0088】この表面層の構成材料と同材料で水平表面を形成し、前記雰囲気ガスと同条件の気体中で前記水平表面上に前記組成Aの溶湯を静かに滴下し、凝固させて凝固物（体積0.01cm<sup>3</sup>）を得、図3または図4に示す方法で前記凝固物の接触角θを測定したところ、95°であった。なお、接触角θの測定は、投影機を用いて光学的に行った。

【0089】また、冷却ロールの回転による冷却ロール周面の最大偏心量ΔRをレーザ変位計により測定したところ、ΔR=10μmであった。

【0090】（実施例2）冷却ロールの表面層（ロール周面）を、Ni-10wt%Ti-10wt%Al-5wt%Mo合金で構成されたもの（表面層の厚さ=5mm）とした以外は、実施例1と同様にして、急冷薄帯を製造した。

【0091】この表面層の構成材料と同材料で水平表面を形成し、前記雰囲気ガスと同条件の気体中で前記水平表面上に前記組成Aの溶湯を静かに滴下し、凝固させて凝固物（体積0.01cm<sup>3</sup>）を得、実施例1と同様の方法で前記凝固物の接触角θを測定したところ、150°であった。

【0092】また、冷却ロールの回転による冷却ロール周面の最大偏心量ΔRをレーザ変位計により測定したところ、ΔR=12μmであった。

【0093】（実施例3）Nd<sub>11</sub>Ce<sub>2</sub>Sm<sub>1</sub>Fe<sub>30</sub>bal. Co<sub>4</sub>Cu<sub>1.5</sub>Ga<sub>1</sub>Ti<sub>0.5</sub>B<sub>6</sub>

（組成B）よりなるインゴットを用いた、同組成の溶湯より急冷薄帯を製造するとともに、冷却ロールの表面層

（ロール周面）を、W-20wt%Zr-3wt%Nb合金で構成されたもの（表面層の厚さ=5mm）とした以外は、実施例1と同様にして、急冷薄帯を製造した。

【0094】この表面層の構成材料と同材料で水平表面を形成し、前記雰囲気ガスと同条件の気体中で前記水平表面上に前記組成Bの溶湯を静かに滴下し、凝固させて凝固物（体積0.01cm<sup>3</sup>）を得、実施例1と同様の方法で前記凝固物の接触角θを測定したところ、70°であった。

【0095】また、冷却ロールの回転による冷却ロール周面の最大偏心量ΔRをレーザ変位計により測定したところ、ΔR=9μmであった。

【0096】＜急冷薄帯の特性評価＞実施例1～3の各急冷薄帯について、その幅wと厚さtとを測定した。この測定は、それぞれ、マイクロスコブにより1つの急冷薄帯につき20箇所の測定点で測定し、これを平均した値とした。

【0097】次に、各急冷薄帯について、TEMによる組織観察結果から、平均結晶粒径を測定するとともに、磁気特性（保磁力iHc、磁気エネルギー積(BH)<sub>max</sub>）を、VSMにより測定した。

【0098】これらの測定結果を下記表1に示す。

【0099】なお、各急冷薄帯の寸法（幅w、厚さt）は、いずれも、測定箇所によるバラツキが極めて少なく（平均値±5%以内）、寸法安定性が高いものであった。

【0100】また、各急冷薄帯について、ロール面を走査型電子顕微鏡（SEM）で観察し、さらに画像解析を行い、この解析結果より、ロール面に対する面積2000μm<sup>2</sup>以上の巨大ディンプルの占める面積率を調べたところ、いずれも、極めて低い値であった。

【0101】

【表1】

急冷薄帯の特性

	接 触 角 θ	急冷薄帯の幅w (mm)	急冷薄帯の厚さt (μm)	平均結晶粒径 (nm)	iHc (MA/m)	(BH) <sub>max</sub> (kJ/m <sup>3</sup> )
実施例1	95°	1.1	26.7	25	0.69	112
実施例2	150°	0.8	30.8	29	0.64	104
実施例3	70°	1.4	24.5	23	0.95	92

【0102】表1からわかるように、実施例1～3の本発明の急冷薄帯は、いずれも、結晶粒の微細化が図れ、高い磁気特性が得られている。

【0103】（実施例4）実施例1の急冷薄帯を粉碎機

（ライカイ機）により不活性ガス中で粉碎して、平均粒径が16μmの磁石粉末とし、この磁石粉末と、エポキシ樹脂2.0wt%と、ヒドラジン系酸化防止剤0.15wt%と、ステアリン酸塩（潤滑剤）0.05wt%とを混

合し、この混合物を十分に混練（120℃×10分）して、ボンド磁石用組成物（コンパウンド）を作製した。

【0104】次いで、このコンパウンドを粉砕して粒状とし、この粒状物を秤量してプレス装置の金型内に充填し、材料温度130℃、圧力6ton/cm<sup>2</sup>で圧縮成形（無磁場中）して成形体を得た。離型後、エポキシ樹脂を加熱硬化させて、直径10mm×高さ7mmの円柱状ボン

ド磁石を得た。  
【0105】（実施例5）実施例2の急冷薄帯を粉砕機（ライカイ機）により不活性ガス中で粉砕して、平均粒

径が20μmの磁石粉末とし、この磁石粉末と、エポキシ樹脂2.5wt%と、ヒドラジン系酸化防止剤0.1wt%と、ステアリン酸塩（潤滑剤）0.1wt%とを混合し、この混合物を十分に混練（120℃×10分）して、ボンド磁石用組成物（コンパウンド）を作製した。

【0106】次いで、このコンパウンドを粉砕して粒状とし、この粒状物を秤量してプレス装置の金型内に充填し、材料温度130℃、圧力6ton/cm<sup>2</sup>で圧縮成形（無磁場中）して成形体を得た。離型後、エポキシ樹脂を加熱硬化させて、直径10mm×高さ7mmの円柱状ボン

ド磁石を得た。  
【0107】（実施例6）実施例3の急冷薄帯を粉砕機（ライカイ機）により不活性ガス中で粉砕して、平均粒

径が18μmの磁石粉末とし、この磁石粉末と、エポキシ樹脂1.9wt%と、ヒドラジン系酸化防止剤0.1wt%と、ステアリン酸塩（潤滑剤）0.05wt%とを混合し、この混合物を十分に混練（120℃×10分）して、ボンド磁石用組成物（コンパウンド）を作製した。

【0108】次いで、このコンパウンドを粉砕して粒状とし、この粒状物を秤量してプレス装置の金型内に充填し、材料温度130℃、圧力6ton/cm<sup>2</sup>で圧縮成形（無磁場中）して成形体を得た。離型後、エポキシ樹脂を加熱硬化させて、直径10mm×高さ7mmの円柱状ボン

ド磁石を得た。  
【0109】（実施例7）実施例4で得た磁石粉末と実施例6で得た磁石粉末とを重量比6：4で均一に混合し、混合磁石粉末を得た。この混合磁石粉末と、エポキシ樹脂2.0wt%と、ヒドラジン系酸化防止剤0.15wt%と、ステアリン酸塩（潤滑剤）0.05wt%とを混

合し、この混合物を十分に混練（120℃×10分）して、ボンド磁石用組成物（コンパウンド）を作製した。

【0110】次いで、このコンパウンドを粉砕して粒状とし、この粒状物を秤量してプレス装置の金型内に充填し、材料温度130℃、圧力6ton/cm<sup>2</sup>で圧縮成形（無磁場中）して成形体を得た。離型後、エポキシ樹脂を加熱硬化させて、直径10mm×高さ7mmの円柱状ボン

ド磁石を得た。  
【0111】（実施例8）実施例4で得た磁石粉末と実施例5で得た磁石粉末と実施例6で得た磁石粉末とを重量比2：3：5で均一に混合し、混合磁石粉末を得た。この混合磁石粉末と、エポキシ樹脂1.8wt%と、ヒドラジン系酸化防止剤0.2wt%と、ステアリン酸（潤滑剤）0.1wt%とを混合し、この混合物を十分に混練（120℃×10分）して、ボンド磁石用組成物（コンパウンド）を作製した。

【0112】次いで、このコンパウンドを粉砕して粒状とし、この粒状物を秤量してプレス装置の金型内に充填し、材料温度130℃、圧力6ton/cm<sup>2</sup>で圧縮成形（無磁場中）して成形体を得た。離型後、エポキシ樹脂を加熱硬化させて、直径10mm×高さ7mmの円柱状ボン

ド磁石を得た。  
【0113】＜ボンド磁石の特性評価＞実施例4～8の各ボンド磁石について、その磁気特性（保磁力*i*<sub>Hc</sub>、磁気エネルギー積(BH)<sub>max</sub>）を、直流自記磁束計により最大印加磁場2MA/mにて測定した。

【0114】さらに、これらのボンド磁石について、60℃×95%RHで500時間までの恒温恒湿試験を行い、耐食性を調べた。この耐食性は、ボンド磁石表面における錆の発生の有無を目視により判別し、錆の発生が全く無かったものを○印、錆の発生が若干認められたものを△印、錆の発生が顕著に認められたものを×印として評価した。

【0115】これらの測定結果を下記表2に示す。また、各ボンド磁石中の磁石粉末の含有量（混合磁石粉末の場合はその総量）を併せて下記表2中に記す。

【0116】

【表2】



ボンド磁石の特性

	用いた磁石粉末	ボンド磁石中の 磁石粉末含有量 (wt%)	iHc (MA/m)	(BH) max (kJ/m <sup>3</sup> )	耐食性
実施例4	実施例1	97.9	0.67	73.5	○
実施例5	実施例2	97.4	0.62	70.6	△~○
実施例6	実施例3	98.0	0.94	62.9	○
実施例7	実施例1、3の混合 (=6:4)	97.9	0.84	70.4	○
実施例8	実施例1、2、3の混合 (=2:3:5)	98.1	0.90	68.5	○

【0117】表2からわかるように、実施例4～8の本発明のボンド磁石は、いずれも、保磁力iHc 0.35 MA/m以上、磁気エネルギー積(BH)max が50 kJ/m<sup>3</sup> 以上と、優れた磁気特性を有しているとともに、耐食性も優れている。

【0118】特に、混合磁石粉末を用いた実施例7および8では、より優れた磁気特性が得られている。

【0119】

【発明の効果】以上述べたように、本発明によれば、適度な濡れ性を有する周面の冷却ロールを用いることにより、溶湯の冷却が良好になされる。そのため、得られた急冷薄帯は、結晶粒の粗大化が防止され、高い磁気特性を持つ。

【0120】特に、急冷薄帯のロール面とフリー面との結晶粒径の差を小さくし、磁気特性の均一化を図ることができる。よって、高機械的強度で優れた磁気特性および耐食性を有する永久磁石を提供することができる。

【0121】また、冷却ロール周面の最大偏心量を小さくすることにより、急冷薄帯の磁気特性のバラツキを有効に防止し、より優れた磁気特性を持つ永久磁石を提供することができる。

【0122】また、本発明では、このような磁石を容易に製造することができ、生産性も高い。

【図面の簡単な説明】

【図1】本発明の磁石材料を製造する装置（急冷薄帯製造装置）の構成例を示す斜視図である。

【図2】図1に示す装置における溶湯の冷却ロールへの衝突部位付近の状態を示す断面側面図である。

【図3】冷却ロール周面の溶湯に対する濡れ性の測定方法を示す断面側面図である。

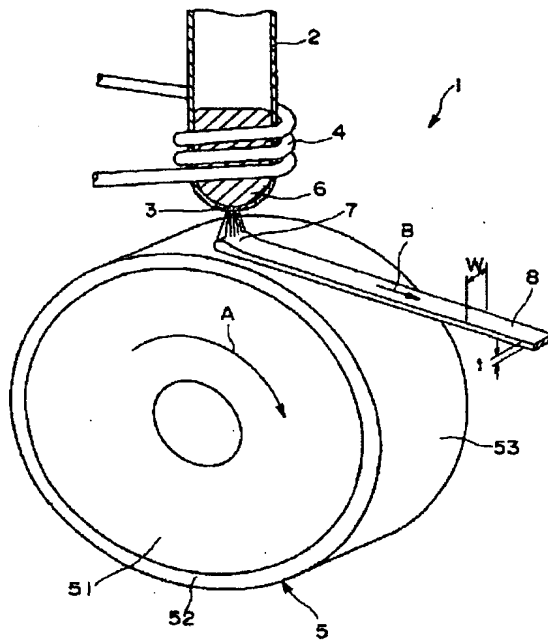
【図4】冷却ロール周面の溶湯に対する濡れ性の測定方法を示す断面側面図である。

【図5】冷却ロールの回転に伴う冷却ロール周面の最大偏心量を示す側面図である。

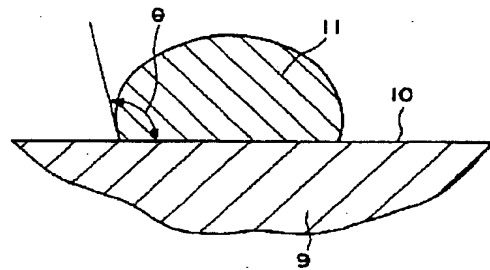
【符号の説明】

- |     |          |
|-----|----------|
| 1   | 急冷薄帯製造装置 |
| 2   | 筒体       |
| 3   | ノズル      |
| 4   | コイル      |
| 5   | 冷却ロール    |
| 51  | 基部       |
| 52  | 表面層      |
| 53  | 周面       |
| 6   | 溶湯       |
| 7   | パドル      |
| 71  | 凝固界面     |
| 8   | 急冷薄帯     |
| 81  | ロール面     |
| 82  | フリー面     |
| 9   | ロール周面材料  |
| 10  | 水平表面     |
| 10' | 面        |
| 11  | 凝固物      |
| 12  | 剝離       |
| 13  | 巨大ディンプル  |

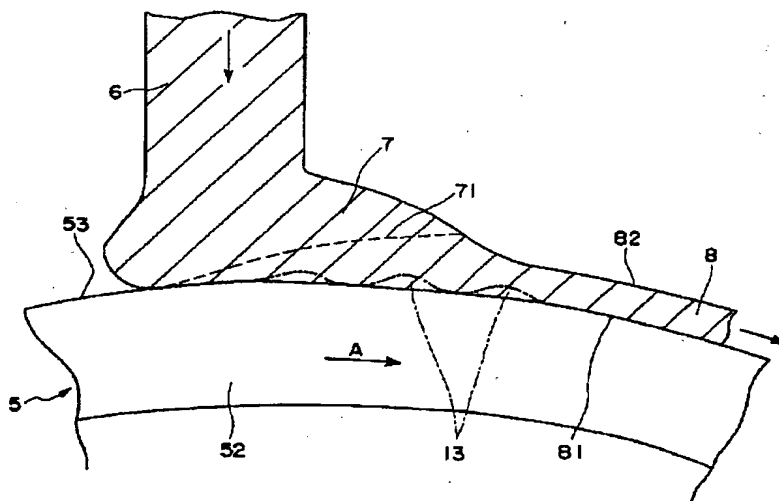
【图 1】



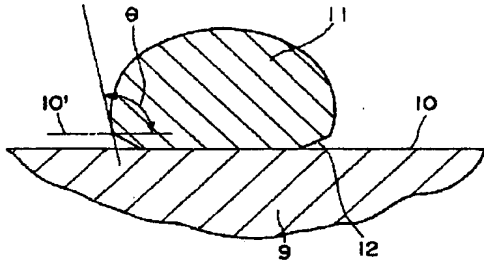
【図 3】



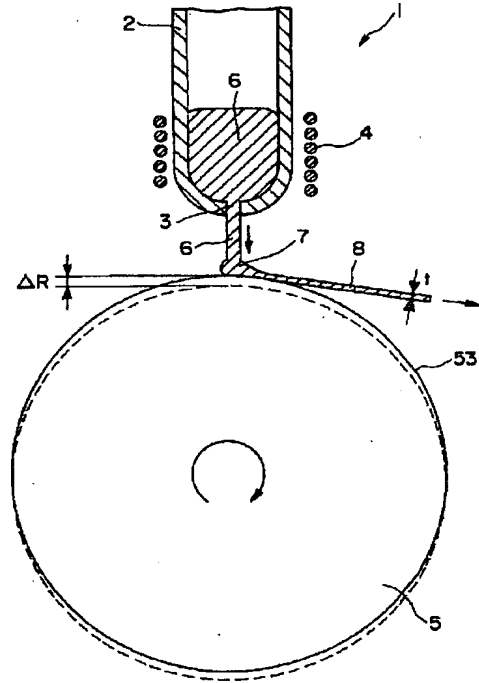
【图 2】



【図 4】



【図 5】



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## PATENT ABSTRACTS OF JAPAN

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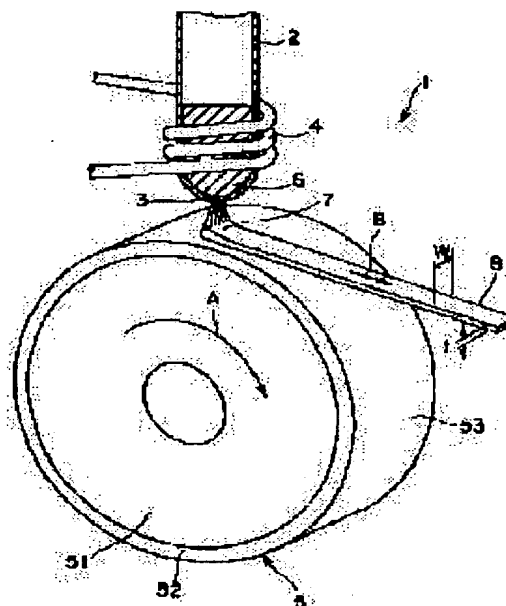
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(72)Inventor : ARAI SEI

**(54) MANUFACTURE OF MAGNET MATERIAL, MAGNET MATERIAL AND BOND MAGNET****(57)Abstract:**

**PROBLEM TO BE SOLVED:** To provide a manufacturing method of magnet material having high magnetic characteristic, a magnet material, and a bond magnet.

**SOLUTION:** A quenched strip manufacturing device 1 is provided with a cylindrical body 2, a heating coil 4, and a cooling roll 5 to be rotated relative to the cylindrical body 2. A nozzle 3 to eject molten metal 6 of a magnet material is formed on a lower end of the cylindrical body 2. In an atmospheric gas, the molten metal 6 is ejected from the nozzle 3, and collided with a circumferential surface 53 of the rotating cooling roll 5, and cooled and solidified to a quenched strip 8. A metallic material to constitute the circumferential surface 53 of the cooling roll 5 has wettability so that the contact angle formed on the horizontal surface of a solidified metal is 70-170° when a droplet of the molten metal 6 is placed and solidified on the horizontal surface of the metallic material.

**LEGAL STATUS**

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## CLAIMS

### [Claim(s)]

[Claim 1] Inject the molten metal of magnet material from a nozzle, make it collide with the peripheral surface of the cooling roller which is rotating to the aforementioned nozzle, and cooling solidification is carried out. The metallic material which is the manufacture method of magnet material of manufacturing a thin band-like magnet material, and constitutes the peripheral surface of the aforementioned cooling roller. The manufacture method of the magnet material characterized by being that from which the contact angle with the aforementioned horizontal surface of the congelation to make becomes 70-170 degrees when the drop of the aforementioned molten metal is made to place and solidify on the horizontal surface of this metallic material.

[Claim 2] Inject the molten metal of magnet material from a nozzle in a controlled atmosphere, make it collide with the peripheral surface of the cooling roller which is rotating to the aforementioned nozzle, and cooling solidification is carried out. The metallic material which is the manufacture method of magnet material of manufacturing a thin band-like magnet material, and constitutes the peripheral surface of the aforementioned cooling roller. The manufacture method of the magnet material characterized by being that from which the contact angle with the aforementioned horizontal surface of the congelation to make becomes 70-170 degrees when the drop of the aforementioned molten metal is made to place and solidify on the horizontal surface of the aforementioned metallic material in the aforementioned controlled atmosphere, a homotypic, and the gas of this \*\*.

[Claim 3] The manufacture method of a magnet material according to claim 1 or 2 that the peripheral velocity of the aforementioned cooling roller is 1-60m/second.

[Claim 4] The manufacture method of a magnet material according to claim 1 to 3 that the maximum eccentricity of the cooling roller peripheral surface accompanying rotation of the aforementioned cooling roller is below the double precision of the average thickness of a thin band-like magnet material obtained.

[Claim 5] The aforementioned controlled atmosphere is the manufacture method of a magnet material according to claim 1 to 4 which is inert gas.

[Claim 6] The aforementioned magnet material is the manufacture method of a magnet material according to claim 1 to 5 which is an alloy containing R (however, at least one sort in the rare earth elements in which R contains Y).

[Claim 7] The aforementioned magnet material is the manufacture method of a magnet material according to claim 1 to 5 which is an alloy containing R (however, at least one sort in the rare earth elements in which R contains Y), and TM (however, TM, at least one sort in transition metals) and B.

[Claim 8] Thin band-like magnet material characterized by being manufactured by the manufacture method of a magnet material according to claim 1 to 7.

[Claim 9] Powdered magnet material characterized by having ground a magnet material according to claim 8, and supposing that it is powdered.

[Claim 10] The bond magnet which combines a powdered magnet material according to claim 9 by the joint resin, and is characterized by the bird clapper.

[Claim 11] The bond magnet according to claim 10 whose content of the magnet material of the shape of aforementioned powder is 82 - 99.5wt%.

[Claim 12] Coercive force  $iH_c$  Bond magnet according to claim 10 or 11 which is 0.35 or more MA/m.

[Claim 13] Magnetic-energy product (BH) max 50 kJ/m<sup>3</sup> Bond magnet according to claim 10 to 12 which it is above.

## DETAILED DESCRIPTION

### [Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the manufacture method, the magnet material, and the bond magnet of magnet material.

[0002]

[Description of the Prior Art] Since the rare earth permanent magnet material which consists of alloys containing rare earth elements as a magnet material has high magnetic properties, when used for a motor etc., it demonstrates high performance.

[0003] Such a magnet material is manufactured by the quenching method which used for example, the quenching thin band manufacturing installation. This manufacture method is as follows.

[0004] Fuse the magnet material (henceforth an "alloy") of predetermined alloy composition, inject the molten metal from a nozzle, and it is made to collide with the peripheral surface of the cooling roller which is rotating to a nozzle, and by making this peripheral surface contact, it quenches, an alloy is solidified and a thin band-like (ribbon base) alloy is formed continuously. This thin band-like alloy is called quenching thin band.

[0005] By the way, although the molten metal injected from the nozzle collides with the peripheral surface of a cooling roller, a paddle (cold slug well) is formed first, and it is cooled after that and solidified, if the cooling rate

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is slow, crystal grain will turn big and rough and magnetic properties will fall.

[0006] Therefore, as a metallic material which constitutes the peripheral surface of a cooling roller, the material which is excellent in thermal conductivity was chosen.

[0007]

[Problem(s) to be Solved by the Invention] However, even when the metallic material excellent in thermal conductivity is used for the peripheral surface of a cooling roller in recent years, the problem that a low case has magnetic properties has arisen.

[0008] The purpose of this invention is to offer the manufacture method, the magnet material, and the bond magnet of the magnet material from which high magnetic properties are obtained.

[0009]

[Means for Solving the Problem] The volumetric flow rate  $Q$  of a quenching thin band (volume of the molten metal injected by per volume = unit time of the quenching thin band manufactured by per unit time) is expressed with the following formula (I) when it is made into the peripheral velocity  $V$  of the width of face  $w$  of a quenching thin band, thickness  $t$ , and a cooling roller.

[0010]  $Q = w \times t \times V \dots (I)$

On the other hand, since a paddle tends to spread more that the wettability (only henceforth "wettability of a roll peripheral surface") to the molten metal of the peripheral surface of a cooling roller is good in latus area on the peripheral surface of a cooling roller, the width of face  $w$  of a quenching thin band becomes large, and if the wettability of a roll peripheral surface is bad, the width of face  $w$  of a quenching thin band will become small conversely.

[0011] Therefore, when a quenching thin band is manufactured having set the peripheral velocity  $V$  of a cooling roller, and the volumetric flow rate  $Q$  of a quenching thin band as constant, when the wettability of the above-mentioned formula (I) to a roll peripheral surface is good, width of face  $w$  is large, a small quenching thin band is obtained for thickness  $t$ , and if the wettability of a roll peripheral surface is conversely bad, width of face  $w$  will be small, a large quenching thin band will be obtained for thickness  $t$ , and it will become things.

[0012] And if thickness  $t$  of a quenching thin band is small, although heat transfer of the thickness direction is made for a short time and is advantageous to detailed-izing of crystal grain If thickness  $t$  of a quenching thin band is large, the heat transfer nature of the thickness direction is bad, the difference of the cooling rate of the roll side (field of the side in contact with the peripheral surface of a cooling roller) of a quenching thin band and a free side (field of the side which does not contact the peripheral surface of a cooling roller) will become large, and crystal grain will especially big-and-rough-become easy to turn into a free side side.

[0013] Since it was such, as a result of inquiring wholeheartedly paying attention to the wettability of a roll peripheral surface, by using the cooling roller which made wettability of a roll peripheral surface the predetermined range, this invention person could attain detailed-ization of crystal grain, found out that outstanding magnetic properties were obtained, and resulted in this invention.

[0014] That is, this invention is as being shown in following the (1) - (13).

[0015] (1) Inject the molten metal of magnet material from a nozzle, make it collide with the peripheral surface of the cooling roller which is rotating to the aforementioned nozzle, and carry out cooling solidification. The metallic material which is the manufacture method of magnet material of manufacturing a thin band-like magnet material, and constitutes the peripheral surface of the aforementioned cooling roller The manufacture method of the magnet material characterized by being that from which the contact angle with the aforementioned horizontal surface of the congelation to make becomes 70-170 degrees when the drop of the aforementioned molten metal is made to place and solidify on the horizontal surface of this metallic material.

[0016] (2) Inject the molten metal of magnet material from a nozzle in a controlled atmosphere, make it collide with the peripheral surface of the cooling roller which is rotating to the aforementioned nozzle, and carry out cooling solidification. The metallic material which is the manufacture method of magnet material of manufacturing a thin band-like magnet material, and constitutes the peripheral surface of the aforementioned cooling roller The manufacture method of the magnet material characterized by being that from which the contact angle with the aforementioned horizontal surface of the congelation to make becomes 70-170 degrees when the drop of the aforementioned molten metal is made to place and solidify on the horizontal surface of the aforementioned metallic material in the aforementioned controlled atmosphere, a homotypic, and the gas of this \*\*.

[0017] (3) The above (1) whose peripheral velocity of the aforementioned cooling roller is 1-60m/second, or the manufacture method of a magnet material given in (2).

[0018] (4) The above (1) whose maximum eccentricity of the cooling roller peripheral surface accompanying rotation of the aforementioned cooling roller is below the double precision of the average thickness of a thin band-like magnet material obtained, or the manufacture method of a magnet material given in either of (3).

[0019] (5) The aforementioned controlled atmosphere is the manufacture method of a magnet material the above (1) which is inert gas, or given in either of (4).

[0020] (6) The aforementioned magnet material is the manufacture method of a magnet material the above (1) which is an alloy containing R (however, at least one sort in the rare earth elements in which R contains Y), or given in either of (5).

[0021] (7) The aforementioned magnet material is the manufacture method of a magnet material the above (1)

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which is an alloy containing R (however, at least one sort in the rare earth elements in which R contains Y), and TM (however, TM, at least one sort in transition metals) and B, or given in either of (5).

[0022] (8) Thin band-like magnet material characterized by being manufactured by the manufacture method of a magnet material the above (1) or given in either of (7).

[0023] (9) Powdered magnet material characterized by having ground the magnet material of a publication to the above (8), and supposing that it is powdered.

[0024] (10) The bond magnet which combines a powdered magnet material of a publication with the above (9) by the joint resin, and is characterized by the bird clapper.

[0025] (11) A bond magnet given in the above (10) whose content of the magnet material of the shape of aforementioned powder is 82 - 99.5wt%.

[0026] (12) Coercive force  $iH_c$  The above (10) which is 0.35 or more MA/m, or bond magnet given in (11).

[0027] (13) Magnetic-energy product (BH) max 50 kJ/m<sup>3</sup> The above (10) which it is above, or bond magnet given in either of (12).

[0028]

[Embodiments of the Invention] Hereafter, the manufacture method, the magnet material, and the bond magnet of the magnet material of this invention are explained in detail, referring to an accompanying drawing.

[0029] The perspective diagram showing the example of composition of the equipment (quenching thin band manufacturing installation) to which drawing 1 manufactures the magnet material of this invention by the single rolling method, and drawing 2 are the cross-section side elevations showing the state near the collision part to the cooling roller of the molten metal in the equipment shown in drawing 1.

[0030] As shown in drawing 1, the quenching thin band manufacturing installation 1 is equipped with the barrel 2 which can contain magnet material, and the cooling roller 5 which rotates in the direction of arrow in drawing A to this barrel 2. The nozzle (orifice) 3 which injects the molten metal of magnet material is formed in the soffit of a barrel 2.

[0031] Moreover, by arranging the coil 4 for heating at an about three nozzle [ of a barrel 2 ] periphery, and impressing a RF to this coil 4, the inside of a barrel 2 is heated (IH) and the magnet material in a barrel 2 is changed into a melting state.

[0032] The cooling roller 5 consists of a base 51 and a surface layer 52 which forms the peripheral surface 53 of a cooling roller 5.

[0033] The component of a base 51 may consist of the quality of the materials which may really consist of the same quality of the materials as a surface layer 52, and are different in a surface layer 52.

[0034] Although especially the component of a base 51 is not limited, it is desirable to consist of metallic materials with high thermal conductivity like copper or a copper system alloy so that the heat of a surface layer 52 can be radiated more quickly.

[0035] Moreover, as for a surface layer 52, it is desirable to consist of metallic materials which are described below.

[0036] Such a quenching thin band manufacturing installation 1 is installed in a chamber (not shown), and operates in the state where it filled up with the controlled atmosphere of inert gas or others preferably in this chamber. In order to prevent oxidization of the quenching thin band 8 especially, as for a controlled atmosphere, it is desirable that it is inert gas.

[0037] As inert gas, although argon gas, gaseous helium, nitrogen gas, etc. are mentioned, for example, especially gaseous helium is desirable. the dimple according to the contamination of a gas stream to the roll side 81 of the quenching thin band 8 when gaseous helium is used for the reason as a controlled atmosphere, especially area -- the huge 2000-micrometer two or more dimples 13 (the imaginary line in drawing 2 shows) -- being generated -- being hard -- it is because heat transfer nature improves and higher magnetic properties are obtained

[0038] When magnet ingredients are paid in a barrel 2, and it heats with a coil 4, it fuses in the quenching thin band manufacturing installation 1 and the molten metal 6 is injected from a nozzle 3, as shown in drawing 2, a molten metal 6 It is cooled quickly, and solidifies, being dragged by the peripheral surface 53 of the rotating cooling roller 5, after colliding with the peripheral surface 53 of a cooling roller 5 and forming a paddle (cold slug well) 7, and the quenching thin band 8 is formed continuously or intermittently. Thus, soon, the roll side 81 separates from a peripheral surface 53, and the formed quenching thin band 8 runs in the direction of arrow B in drawing 1. In addition, a dotted line shows the solidification interface 71 of a molten metal among drawing 2.

[0039] Although the suitable range changes with wettability to composition of an alloy molten metal, and the molten metal 6 of a peripheral surface 53 etc., usually, as for the peripheral velocity V of a cooling roller 5, it is desirable that it is 1-60m/second, and it is more desirable that it is 5-40m/second. If the peripheral velocity of a cooling roller 5 is too slow, thickness t of the quenching thin band 8 will become thick with the volumetric flow rate Q of the quenching thin band 8 (refer to the aforementioned formula (I)), the diameter of crystal grain will increase, if the peripheral velocity V of a cooling roller 5 is too quick conversely, it will become amorphous and, in any case, magnetic properties will fall.

[0040] The metallic material which constitutes the peripheral surface 53 of a cooling roller 5, i.e., the metallic material which constitutes a surface layer 52, (henceforth "roll peripheral surface material") has the wettability

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(only henceforth "wettability") to the following molten metals 6, and it is constituted. That is, as shown in drawing 3, a horizontal surface 10 is formed with the roll peripheral surface material 9, the drop of a molten metal 6 is placed on this horizontal surface 10, and when it is made to solidify, the contact angle  $\theta$  with the horizontal surface 10 of the congelation 11 to make becomes 70-170 degrees. In this case, it is desirable that it is 80-165 degrees, as for a contact angle  $\theta$ , it is more desirable that it is 90-160 degrees, and it is still more desirable that it is 95-150 degrees.

[0041] The wettability of the peripheral surface 53 of a cooling roller 5 is not measured directly, but a horizontal surface 10 is formed with the same material (roll peripheral surface material) as it here, and the wettability of this horizontal surface 10 is measured because it cannot stop and put the drop of a molten metal 6 on a fixed position but measurement of a contact angle is impossible or difficult for it, since a peripheral surface 53 is a curve convex.

[0042] In addition, in order to obtain more the correspondence relation with wettability of the peripheral surface 53 of the cooling roller 5 used for actual manufacture of a quenching thin band to accuracy on the occasion of measurement of a contact angle  $\theta$ , it is desirable to perform solidification of the drop of a molten metal 6 in the aforementioned controlled atmosphere, a homotypic, and the gas of this \*\*. Moreover, the volume of a congelation 11 is 3 0.005-0.1cm. Measuring in the range is desirable.

[0043] If a contact angle  $\theta$  exceeds the upper limit of the above-mentioned range, the wettability of a peripheral surface 53 will be bad, and it will become the inclination for thickness  $t$  of the quenching thin band 8 to become thick with the volumetric flow rate  $Q$  of the quenching thin band 8, especially the crystal grain by the side of the free side 82 of the quenching thin band 8 will turn big and rough, and magnetic properties will fall. In addition, if a volumetric flow rate  $Q$  is made small also by this case, although thickness  $t$  also becomes thin (refer to the aforementioned formula (I)) and this fault is canceled or eased, since the fall of productivity is caused, it is not desirable.

[0044] Moreover, since the wettability of a peripheral surface 53 is too good in a contact angle  $\theta$  being under the lower limit of the above-mentioned range, a paddle 7 spreads too much, therefore the configuration of the quenching thin band 8 and a size (width of face  $w$ , thickness  $t$ ) become unstable, and the uniform and homogeneous quenching thin band 8 is not obtained (variation arises in a state, magnetic properties, etc. of crystal grain).

[0045] In addition, on the occasion of measurement of a contact angle  $\theta$ , the exfoliation (coming floating) 12 as shown in drawing 4 may arise by the solidification shrinkage near the solidification interface the drop of a molten metal 6 contacts a horizontal surface 10. In this case, the portion which exfoliation 12 produced is excepted and a contact angle  $\theta$  is measured. Namely, a contact angle  $\theta$  is measured by using as datum level field 10' parallel to the horizontal surface 10 which passes along the upper limit (peak) of exfoliation 12.

[0046] By the way, in the quenching thin band manufacturing installation 1, a cooling roller 5 faces rotating, and as shown in drawing 5, some eccentricity (axial deflection) arises from the dimensional accuracy (roundness) of cooling roller 5 the very thing, the installation precision over the bearing of a cooling roller 5, etc.

[0047] If this eccentricity is large, the front face and the solidification interface 71 of a melting alloy in a paddle 7 vibrate, change will arise in the size (width of face  $w$ , thickness  $t$ ) of the obtained quenching thin band 8, or change will arise at the time when the roll side 81 of the quenching thin band 8 touches the peripheral surface 53 of a cooling roller 5. Furthermore, the incidence rate of the aforementioned huge dimple 13 also increases. Consequently, the cooling rate of the quenching thin band 8 etc. is changed, and variation arises in magnetic properties. And magnetic properties fall [ the bond magnet using the magnet powder or it which were obtained from such a quenching thin band 8 ].

[0048] In order to prevent such a thing, it is desirable to make maximum eccentricity  $\Delta R$  (to refer to drawing 5) of the peripheral surface 53 of the cooling roller 5 accompanying rotation of a cooling roller 5 below into the double precision of thickness (average)  $t$  of the quenching thin band 8 which can be obtained by this invention, considering as 1.5 or less times is more desirable, and considering as 1 or less time is still more desirable. Thereby, the magnetic properties of the obtained quenching thin band 8 can be made more into homogeneity. And the magnetic properties of the bond magnet manufactured from this can be raised. Especially in this invention, further excellent magnetic properties can be demonstrated according to the synergistic effect of specifying such maximum eccentricity  $\Delta R$  and specifying the wettability of the peripheral surface 53 mentioned above.

[0049] Here, especially the lower limit of maximum eccentricity  $\Delta R$  is the limitation of the precision of the bearing which supports the limitation of the process tolerance of the peripheral surface 53 of a cooling roller 5, and a cooling roller 5 although not limited to 0.1 micrometers. It can consider as a grade.

[0050] In addition, maximum eccentricity  $\Delta R$  can be measured with precision sizer vessels, such as for example, a laser displacement gage, an electrostatic displacement gage, and a precision gage.

[0051] As a magnet material in this invention, it is R (however, R). The alloy containing at least one sort in the rare earth elements containing Y, especially R (however, R) Rare earth permanent magnet material like the alloy containing at least one sort in the rare earth elements containing Y, and TM (however, TM, at least one sort in transition metals) and B is mentioned, and the thing of composition of following [1] - [4] is desirable.

[0052] [1] What makes a fundamental component the rare earth elements which are mainly concerned with Sm, and the transition metals which are mainly concerned with Co (henceforth a Sm-Co system alloy).

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[0053] [2] What makes a fundamental component the transition metals which are mainly concerned with R (however, at least one sort in the rare earth elements in which R contains Y), and Fe, and B (henceforth a R-Fe-B system alloy).

[0054] [3] What makes a fundamental component the rare earth elements which are mainly concerned with Sm, the transition metals which are mainly concerned with Fe, and the element between grids which is mainly concerned with N (henceforth an Sm-Fe-N system alloy).

[4] What makes a fundamental component transition metals, such as R (at least one sort however, among the rare earth elements in which R contains Y), and Fe, and has a magnetic phase on nano meter level (nano crystal magnet).

[0055] As a typical thing of a Sm-Co system alloy, SmCo5 and Sm2TM17 (however, TM, transition metals) are mentioned.

[0056] As a typical thing of a R-Fe-B system alloy, a Nd-Fe-B system alloy, a Pr-Fe-B system alloy, a Nd-Pr-Fe-B system alloy, a Ce-Nd-Fe-B system alloy, a Ce-Pr-Nd-Fe-B system alloy, the thing that replaced a part of Fe in these by other transition metals, such as Co and nickel, are mentioned.

[0057] It is Sm2Fe17N3 which nitrated and produced Sm2Fe17 alloy as a typical thing of an Sm-Fe-N system alloy. It is mentioned.

[0058] as the aforementioned rare earth elements, Y, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, and a misch metal mention -- having -- these -- one sort -- or two or more sorts can be included moreover, Fe, Co, nickel, etc. mention as the aforementioned transition metals -- having -- these -- one sort -- or two or more sorts can be included Moreover, in order to raise magnetic properties, in magnet material, B, aluminum, Cu, Ga, Si, Ti, V, Ta, Zr, Nb, Mo, Hf, Ag, Zn, P, germanium, etc. can also be contained if needed.

[0059] The magnetic properties in which the quenching thin band (thin band-like magnet material) 8 of this invention obtained by the above manufacture methods turned minutely, consequently crystal grain was excellent are obtained.

[0060] Moreover, a powdered magnet material (magnet powder) of this invention is obtained by grinding such a quenching thin band 8.

[0061] Especially the method of pulverization is not limited, for example, can be performed using various pulverization equipments, such as a ball mill, a vibration mill, a jet mill, and a pin mill, and a shredding equipment. In this case, pulverization can also be performed in a non-oxidizing atmosphere like [ under a vacuum or a reduced pressure state (for example,  $1 \times 10^{-1}$  -  $1 \times 10^{-6}$  Torr) or in inert gas, such as nitrogen gas, argon gas, and gaseous helium, ], in order to prevent oxidization.

[0062] A different thing only not only in the thing of the same composition which mixed the magnet powder of two or more sorts of composition is sufficient as such magnet powder. For example, what mixed at least two sorts in the thing of composition of aforementioned [1] - [4] is mentioned. In this case, it can have the advantage of each magnet powder to mix simultaneously, and more excellent magnetic properties can be obtained easily.

[0063] Moreover, in the case of the thing for manufacturing the bond magnet mentioned later, although not limited, especially the mean particle diameter of magnet powder is 0.5-60 micrometers. A grade is desirable and it is 1-40 micrometers. A grade is more desirable. moreover, in order to obtain the good moldability at the time of fabrication by a small amount of joint resin which is mentioned later, the particle size distribution of magnet powder is distributed to some extent -- \*\*\*\*'s (there is variation) -- it is desirable The rate of a hole of the obtained bond magnet can be reduced by this, the mechanical strength of a bond magnet can be raised more, and magnetic properties can be improved further.

[0064] In addition, the mean particle diameters may differ for every composition of different magnet powder case [ powder ] and mixed although the magnet powder of two or more sorts of composition was mixed. Moreover, what is necessary is to just be manufactured by the method of a different this invention which at least one sort in the magnet powder of two or more sorts of composition mentioned above the case in the end of such mixed powder.

[0065] When a bond magnet is manufactured using the above magnet powder, such magnet powder has good unity (wettability of a joint resin) with a joint resin, therefore this bond magnet has a high mechanical strength and it becomes the thing excellent in thermal stability (thermal resistance) and corrosion resistance. Therefore, the magnet powder concerned fits manufacture of a bond magnet.

[0066] In addition, the magnet powder (powdered magnet material) of this invention cannot be overemphasized by that you may be what it is not limited to what is used for manufacture of a bond magnet, for example, is used for manufacture of a sintered magnet.

[0067] Next, the bond magnet of this invention is explained.

[0068] The bond magnet of this invention comes to join the above-mentioned magnet powder together by the joint resin.

[0069] As a joint resin (binder), any of thermoplastics and thermosetting resin are sufficient.

[0070] As thermoplastics, for example A polyamide (example : nylon 6, Nylon 46, Nylon 66, Nylon 610, Nylon 612, Nylon 11, Nylon 12, Nylon 612, nylon 6-66), Liquid crystal polymers, such as a thermoplastic polyimide and an aromatic polyester, a polyphenylene oxide, Polyolefines, such as polyphenylene sulfide, polyethylene, polypropylene, and an ethylene vinylacetate copolymer, A denaturation polyolefine, a polycarbonate, a polymethylmethacrylate, Polyester, such as a polyethylene terephthalate and a polybutylene terephthalate,

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The copolymer which is mainly concerned with these, a blend object, a polymer alloy, etc. are mentioned, and a polyether, a polyether ether ketone, polyether imide, a polyacetal, etc. can mix and use 1 of sorts of these, and two sorts or more.

[0071] Also among these, especially the moldability is excellent, and since the mechanical strength is high, that which is mainly concerned with a liquid crystal polymer and polyphenylene sulfide from the point of a polyamide and heat-resistant improvement is desirable. Moreover, these thermoplastics is excellent also in kneading nature with magnet powder.

[0072] Such thermoplastics has the advantage that selection wide range like what thought the moldability as important, and the thing which thought thermal resistance and the mechanical strength as important is attained by the kind, copolymerization-ization, etc.

[0073] On the other hand, as thermosetting resin, various epoxy resins, such as a bisphenol type, a novolak type and a naphthalene system, phenol resin, a urea resin, melamine resin, a polyester (unsaturated polyester) resin polyimide resin, silicone resin, a polyurethane resin, etc. are mentioned, and 1 of sorts of these and two sorts or more can be mixed and used, for example.

[0074] From the point of especially the moldability being excellent also among these, and a mechanical strength being high and excelling in thermal resistance, an epoxy resin, phenol resin, polyimide resin, and silicone resin are desirable, and especially an epoxy resin is desirable. Moreover, these thermosetting resin is excellent also in kneading nature with magnet powder, and the homogeneity of kneading.

[0075] In addition, the thing of a solid [ thing / liquefied ] (powdered) is sufficient as the thermosetting resin (un-hardening) used at a room temperature.

[0076] Such a bond magnet of this invention is manufactured as follows, for example. Magnet powder, a joint resin, and the constituent for bond magnets (compound) that contains additives (an antioxidant, lubricant, etc.) if needed are manufactured, and it fabricates in a desired magnet configuration all over a magnetic field or a non-magnetic field by methods, such as compression molding, extrusion molding, and injection molding, using this constituent for bond magnets. When a joint resin is thermosetting resin, it is hardened by heating etc. after fabrication.

[0077] As for the content of the magnet powder in a bond magnet, it is desirable that it is about 82-99.5wt%, and it is more desirable that it is about 90-99wt%. Although the bond magnet was especially manufactured by compression molding, as for the content of magnet powder, to a case, it is desirable that it is about 93-99.5wt%, and it is more desirable to it that it is about 95-99wt%.

[0078] If improvement in magnetic properties (especially magnetic-energy product) cannot be aimed at if there are too few contents of magnet powder, and there are too many contents of magnet powder, joint resins content will decrease relatively and a moldability will fall.

[0079] Such a bond magnet of this invention demonstrates outstanding magnetic properties from the property of the quenching thin band 8 used as the raw material mentioned above, the numerousness of the manufacture conditions of a bond magnet, and the contents of the magnet powder contained in a bond magnet, etc.

[0080] That is, the bond magnet of this invention is coercive force  $iH_c$ . 0.35 or more MA/m is 0.50 or more MA/m more preferably.

[0081] The bond magnet of this invention, especially the bond magnet fabricated all over the non-magnetic field are magnetic-energy (product BH) max. It is 50 kJ/m<sup>3</sup> preferably. It is 70 kJ/m<sup>3</sup> more preferably above. It is above.

[0082] It is not limited, for example, about a configuration, the thing of all configurations, such as the shape of the shape of a pillar, a prismatic, and a cylinder (the shape of a ring), circular, plate-like, and a curve tabular, is possible for the configuration of the bond magnet of this invention, especially a size, etc., for example, and the thing of all sizes is possible for them from a thing also with the large-sized size to a micro thing.

[0083]

[Example] Hereafter, the concrete example of this invention is explained.

[0084] (Example 1) The hardener ingot as which alloy composition is expressed in Nd<sub>10</sub>Pr<sub>2.5</sub> Fe<sub>bal</sub>.Co<sub>6</sub> aluminum<sub>3</sub> Cu<sub>1.5</sub> Nb<sub>1</sub> Ga<sub>1</sub> B<sub>5</sub> (composition A) was cast. About 15g sample was started from this ingot.

[0085] The quenching thin band manufacturing installation 1 of composition of being shown in drawing 1 was prepared, and the aforementioned sample was put in in the quartz tube which prepared the nozzle (circular hole orifice) in the bottom. After deaerating the inside of the chamber by which the quenching thin band manufacturing installation 1 is contained, gaseous helium was introduced as a controlled atmosphere and it considered as the temperature of 21 degrees C, and the controlled atmosphere of pressure 60KPa.

[0086] Then, the ingot sample in a quartz tube was fused by high-frequency induction heating, this molten metal was injected by the differential pressure of the internal pressure of a quartz tube, and an ambient pressure towards the peripheral surface of the cooling roller with a diameter [ of 200mm ], and a width of face of 20mm which rotates by 1500rpm (peripheral velocity : 15.7m/(second)), and the quenching thin band of the alloy of the aforementioned composition A was obtained.

[0087] The surface layer (roll peripheral surface) of a cooling roller should consist of Pd-8wt%Ru-2wt%Pt alloys. Moreover, thickness of this surface layer was set to 5mm.

[0088] It was 95 degrees when the contact angle theta of the aforementioned congelation was measured by the method which forms a horizontal surface with the component and this material of this surface layer, trickles

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the molten metal of the aforementioned composition A calmly, is made to solidify it on the aforementioned horizontal surface in the gas of the aforementioned controlled atmosphere and these conditions, obtains a congelation (volume 0.01cm<sup>3</sup>), and is shown in drawing 3 or drawing 4. In addition, measurement of a contact angle theta was optically performed using the projector.

[0089] moreover, the place which measured maximum eccentricity deltaR of the cooling roller peripheral surface by rotation of a cooling roller by the laser displacement gage -- deltaR=10micrometer it was.

[0090] (Example 2) The quenching thin band was manufactured like the example 1 except having constituted the surface layer (roll peripheral surface) of a cooling roller from a nickel-10wt%Ti-10wt%aluminum-5wt%Mo alloy.

[0091] It was 150 degrees, when the horizontal surface was formed with the component and this material of this surface layer, the molten metal of the aforementioned composition A is dropped calmly, was made to solidify on the aforementioned horizontal surface in the gas of the aforementioned controlled atmosphere and these conditions, the congelation (volume 0.01cm<sup>3</sup>) was obtained and the contact angle theta of the aforementioned congelation was measured by the same method as an example 1.

[0092] moreover, the place which measured maximum eccentricity deltaR of the cooling roller peripheral surface by rotation of a cooling roller by the laser displacement gage -- deltaR=12micrometer it was.

[0093] (Example 3) While manufacturing a quenching thin band from the molten metal of this composition which used the ingot which consists of Nd11Ce2 Sm1 Febal.Co4 Cu1.5 Ga1 Ti0.5 B6 (composition B) The quenching thin band was manufactured like the example 1 except having constituted the surface layer (roll peripheral surface) of a cooling roller from a W-20wt%Zr-3wt%Nb alloy.

[0094] It was 70 degrees, when the horizontal surface was formed with the component and this material of this surface layer, the molten metal of the aforementioned composition B is dropped calmly, was made to solidify on the aforementioned horizontal surface in the gas of the aforementioned controlled atmosphere and these conditions, the congelation (volume 0.01cm<sup>3</sup>) was obtained and the contact angle theta of the aforementioned congelation was measured by the same method as an example 1.

[0095] moreover, the place which measured maximum eccentricity deltaR of the cooling roller peripheral surface by rotation of a cooling roller by the laser displacement gage -- deltaR=9micrometer it was.

[0096] About each quenching thin band of the <characterization of quenching thin band> examples 1-3, the width of face w and thickness t were measured. This measurement was measured by 20 point of measurement per quenching thin band by the microscope, and was made into the value which averaged this, respectively.

[0097] Next, while measuring the diameter of average crystal grain from the organization observation result by transverse electromagnetic about each quenching thin band, magnetic properties (coercive force iHc and magnetic-energy (product BH) max) were measured by VSM.

[0098] These measurement results are shown in the following table 1.

[0099] In addition, there was very little variation by the measurement part (less than \*\*5% of averages), and each size (width of face w, thickness t) of each quenching thin band had high dimensional stability.

[0100] Moreover, when the rate of area which observes a roll side with a scanning electron microscope (SEM), and performs image analysis further about each quenching thin band, and the with a 2000-micrometer area [or more 2] huge dimple to a roll side occuppies from this analysis result was investigated, all were very low values.

[0101]

[Table 1]

急冷薄帯の特性

	接 触 角 $\theta$	急冷薄帯の幅w (mm)	急冷薄帯の厚さt ( $\mu$ m)	平均結晶粒径 (nm)	iHc (MA/m)	(BH) max (kJ/m <sup>3</sup> )
実施例1	95°	1.1	26.7	25	0.69	112
実施例2	150°	0.8	30.9	29	0.64	104
実施例3	70°	1.4	24.5	23	0.95	92

[0102] As shown in Table 1, each quenching thin band of this invention of examples 1-3 can attain detailed-ization of crystal grain, and high magnetic properties are obtained.

[0103] (Example 4) A grinder (RAIKAI machine) grinds the quenching thin band of an example 1 in inert gas, and a mean particle diameter is 16 micrometers. It considered as magnet powder, this magnet powder, epoxy resin 2.0wt%, hydrazine system antioxidant 0.15wt%, and stearate (lubricant) 0.05wt% were mixed, this mixture was fully kneaded (120 degree-Cx 10 minutes), and the constituent for bond magnets (compound) was

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produced.

[0104] subsequently -- grinding this compound and being granular -- carrying out -- this granular object -- weighing capacity -- carrying out -- the metal mold of press equipment -- inside -- being filled up -- 130 degrees C of material temperature, and pressure 6 ton/cm<sup>2</sup> It pressed (inside of a non-magnetic field), and the Plastic solid was obtained. Heat hardening of the epoxy resin was carried out after mold release, and the pillar-like bond magnet with a diameter [ of 10mm ] x height of 7mm was obtained.

[0105] (Example 5) A grinder (RAIKAI machine) grinds the quenching thin band of an example 2 in inert gas, and a mean particle diameter is 20 micrometers. It considered as magnet powder, this magnet powder, epoxy resin 2.5wt%, hydrazine system antioxidant 0.1wt%, and stearate (lubricant) 0.1wt% were mixed, this mixture was fully kneaded (120 degree-Cx 10 minutes), and the constituent for bond magnets (compound) was produced

[0106] subsequently -- grinding this compound and being granular -- carrying out -- this granular object -- weighing capacity -- carrying out -- the metal mold of press equipment -- inside -- being filled up -- 130 degrees C of material temperature, and pressure 6 ton/cm<sup>2</sup> It pressed (inside of a non-magnetic field), and the Plastic solid was obtained. Heat hardening of the epoxy resin was carried out after mold release, and the pillar-like bond magnet with a diameter [ of 10mm ] x height of 7mm was obtained.

[0107] (Example 6) A grinder (RAIKAI machine) grinds the quenching thin band of an example 3 in inert gas, and a mean particle diameter is 18 micrometers. It considered as magnet powder, this magnet powder, epoxy resin 1.9wt%, hydrazine system antioxidant 0.1wt%, and stearate (lubricant) 0.05wt% were mixed, this mixture was fully kneaded (120 degree-Cx 10 minutes), and the constituent for bond magnets (compound) was produced.

[0108] subsequently -- grinding this compound and being granular -- carrying out -- this granular object -- weighing capacity -- carrying out -- the metal mold of press equipment -- inside -- being filled up -- 130 degrees C of material temperature, and pressure 6 ton/cm<sup>2</sup> It pressed (inside of a non-magnetic field), and the Plastic solid was obtained. Heat hardening of the epoxy resin was carried out after mold release, and the pillar-like bond magnet with a diameter [ of 10mm ] x height of 7mm was obtained.

[0109] (Example 7) The magnet powder obtained in the example 4 and the magnet powder obtained in the example 6 were uniformly mixed by the weight ratio 6:4, and mixed magnet powder was obtained. This mixed magnet powder, epoxy resin 2.0wt%, hydrazine system antioxidant 0.15wt%, and stearate (lubricant) 0.05wt% were mixed, this mixture was fully kneaded (120 degree-Cx 10 minutes), and the constituent for bond magnets (compound) was produced.

[0110] subsequently -- grinding this compound and being granular -- carrying out -- this granular object -- weighing capacity -- carrying out -- the metal mold of press equipment -- inside -- being filled up -- 130 degrees C of material temperature, and pressure 6 ton/cm<sup>2</sup> It pressed (inside of a non-magnetic field), and the Plastic solid was obtained. Heat hardening of the epoxy resin was carried out after mold release, and the pillar-like bond magnet with a diameter [ of 10mm ] x height of 7mm was obtained.

[0111] (Example 8) The magnet powder obtained in the example 4, the magnet powder obtained in the example 5, and the magnet powder obtained in the example 6 were uniformly mixed by the weight ratio 2:3:5, and mixed magnet powder was obtained. This mixed magnet powder, epoxy resin 1.8wt%, hydrazine system antioxidant 0.2wt%, and stearin acid (lubricant) 0.1wt% were mixed, this mixture was fully kneaded (120 degree-Cx 10 minutes), and the constituent for bond magnets (compound) was produced.

[0112] subsequently -- grinding this compound and being granular -- carrying out -- this granular object -- weighing capacity -- carrying out -- the metal mold of press equipment -- inside -- being filled up -- 130 degrees C of material temperature, and pressure 6 ton/cm<sup>2</sup> It pressed (inside of a non-magnetic field), and the Plastic solid was obtained. Heat hardening of the epoxy resin was carried out after mold release, and the pillar-like bond magnet with a diameter [ of 10mm ] x height of 7mm was obtained.

[0113] About each bond magnet of the <characterization of bond magnet> examples 4-8, the magnetic properties (coercive force iH<sub>c</sub> and magnetic energy (product BH) max) were measured in maximum impression magnetic field 2 MA/m with the account fluxmeter of \*\*\*\*\*.

[0114] furthermore -- these bond magnets -- 60 degree-Cx95%RH -- the constant temperature by 500 hours -- the constant humidity examination was performed and corrosion resistance was investigated This corrosion resistance distinguished the existence of generating of the rust in a bond magnet front face by viewing, and evaluated as a x mark what \*\* mark and generating of rust were notably accepted in in what O mark and generating of rust were accepted in a little in the thing without generating of rust.

[0115] These measurement results are shown in the following table 2. Moreover, the content (in the case of mixed magnet powder, it is the total amount) of the magnet powder in each bond magnet is collectively described all over the following table 2.

[0116]

[Table 2]

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ボンド磁石の特性

	用いた磁石粉末	ボンド磁石中の 磁石粉末含有量 (wt%)	i Hc (MA/m)	(BH) max (kJ/m <sup>3</sup> )	耐食性
実施例4	実施例1	97.9	0.67	73.5	○
実施例5	実施例2	97.4	0.62	70.6	△~○
実施例6	実施例3	98.0	0.94	62.9	○
実施例7	実施例1、3の混合 (=6:4)	97.9	0.84	70.4	○
実施例8	実施例1、2、3の混合 (=2:3:5)	98.1	0.90	68.5	○

[0117] As shown in Table 2, each bond magnet of this invention of examples 4-8 is more than coercive force iHc 0.35 MA/m and magnetic-energy (product BH) max. 50 kJ/m<sup>3</sup> Corrosion resistance is also excellent while having the magnetic properties which were excellent with the above.

[0118] Especially, more excellent magnetic properties are obtained in the examples 7 and 8 using mixed magnet powder.

[0119]

[Effect of the Invention] Cooling of a molten metal is made good by using the cooling roller of a peripheral surface which was described above and which has moderate wettability like according to this invention. Therefore, big and rough-ization of crystal grain is prevented, and the obtained quenching thin band has high magnetic properties.

[0120] Especially, the difference of the diameter of crystal grain of the roll side of a quenching thin band and a free side can be made small, and equalization of magnetic properties can be attained. Therefore, the permanent magnet which has magnetic properties and corrosion resistance excellent in the high mechanical strength can be offered.

[0121] Moreover, by making small the maximum eccentricity of a cooling roller peripheral surface, the variation in the magnetic properties of a quenching thin band can be prevented effectively, and a permanent magnet with more excellent magnetic properties can be offered.

[0122] Moreover, in this invention, such a magnet can be manufactured easily, and productivity is also high.

## TECHNICAL FIELD

[The technical field to which invention belongs] this invention relates to the manufacture method, the magnet material, and the bond magnet of magnet material.

## PRIOR ART

[Description of the Prior Art] Since the rare earth permanent magnet material which consists of alloys containing rare earth elements as a magnet material has high magnetic properties, when used for a motor etc., it demonstrates high performance.

[0003] Such a magnet material is manufactured by the quenching method which used for example, the quenching thin band manufacturing installation. This manufacture method is as follows.

[0004] Fuse the magnet material (henceforth an "alloy") of predetermined alloy composition, inject the molten metal from a nozzle, and it is made to collide with the peripheral surface of the cooling roller which is rotating to a nozzle, and by making this peripheral surface contact, it quenches, an alloy is solidified and a thin band-like (ribbon base) alloy is formed continuously. This thin band-like alloy is called quenching thin band.

[0005] By the way, although the molten metal injected from the nozzle collides with the peripheral surface of a cooling roller, a paddle (cold slug well) is formed first, and it is cooled after that and solidified, if the cooling rate is slow, crystal grain will turn big and rough and magnetic properties will fall.

[0006] Therefore, as a metallic material which constitutes the peripheral surface of a cooling roller, the material which is excellent in thermal conductivity was chosen.

## EFFECT OF THE INVENTION

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[Effect of the Invention] Cooling of a molten metal is made good by using the cooling roller of a peripheral surface which was described above and which has moderate wettability like according to this invention. Therefore, big and rough-ization of crystal grain is prevented, and the obtained quenching thin band has high magnetic properties.

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## TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] However, even when the metallic material excellent in thermal conductivity is used for the peripheral surface of a cooling roller in recent years, the problem that magnetic properties may be low has arisen.

[0008] The purpose of this invention is to offer the manufacture method, the magnet material, and the bond magnet of the magnet material from which high magnetic properties are obtained.

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## MEANS

[Means for Solving the Problem] The volumetric flow rate  $Q$  of a quenching thin band (volume of the molten metal injected by per volume = unit time of the quenching thin band manufactured by per unit time) is expressed with the following formula (I) when it is made into the peripheral velocity  $V$  of the width of face  $w$  of a quenching thin band, thickness  $t$ , and a cooling roller.

[0010]  $Q = w \times t \times V \dots (I)$

On the other hand, since a paddle tends to spread more that the wettability (only henceforth "wettability of a roll peripheral surface") to the molten metal of the peripheral surface of a cooling roller is good in latus area on the peripheral surface of a cooling roller, the width of face  $w$  of a quenching thin band becomes large, and if the wettability of a roll peripheral surface is bad, the width of face  $w$  of a quenching thin band will become small conversely.

[0011] Therefore, when a quenching thin band is manufactured having set the peripheral velocity  $V$  of a cooling roller, and the volumetric flow rate  $Q$  of a quenching thin band as constant, when the wettability of the above-mentioned formula (I) to a roll peripheral surface is good, width of face  $w$  is large, a small quenching thin band is obtained for thickness  $t$ , and if the wettability of a roll peripheral surface is conversely bad, width of face  $w$  will be small, a large quenching thin band will be obtained for thickness  $t$ , and it will become things.

[0012] And if thickness  $t$  of a quenching thin band is small, although heat transfer of the thickness direction is made for a short time and is advantageous to detailed-izing of crystal grain If thickness  $t$  of a quenching thin band is large, the heat transfer nature of the thickness direction is bad, the difference of the cooling rate of the roll side (field of the side in contact with the peripheral surface of a cooling roller) of a quenching thin band and a free side (field of the side which does not contact the peripheral surface of a cooling roller) will become large, and crystal grain will especially big-and-rough-become easy to turn into a free side side.

[0013] Since it was such, as a result of inquiring wholeheartedly paying attention to the wettability of a roll peripheral surface, by using the cooling roller which made wettability of a roll peripheral surface the predetermined range, this invention person could attain detailed-ization of crystal grain, found out that outstanding magnetic properties were obtained, and resulted in this invention.

[0014] That is, this invention is as being shown in following the (1) - (13).

[0015] (1) Inject the molten metal of magnet material from a nozzle, make it collide with the peripheral surface of the cooling roller which is rotating to the aforementioned nozzle, and carry out cooling solidification. The metallic material which is the manufacture method of magnet material of manufacturing a thin band-like magnet material, and constitutes the peripheral surface of the aforementioned cooling roller The manufacture method of the magnet material characterized by being that from which the contact angle with the aforementioned horizontal surface of the congelation to make becomes 70-170 degrees when the drop of the aforementioned molten metal is made to place and solidify on the horizontal surface of this metallic material.

[0016] (2) Inject the molten metal of magnet material from a nozzle in a controlled atmosphere, make it collide with the peripheral surface of the cooling roller which is rotating to the aforementioned nozzle, and carry out cooling solidification. The metallic material which is the manufacture method of magnet material of

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manufacturing a thin band-like magnet material, and constitutes the peripheral surface of the aforementioned cooling roller. The manufacture method of the magnet material characterized by being that from which the contact angle with the aforementioned horizontal surface of the congelation to make becomes 70-170 degrees when the drop of the aforementioned molten metal is made to place and solidify on the horizontal surface of the aforementioned metallic material in the aforementioned controlled atmosphere, a homotypic, and the gas of this \*\*.

[0017] (3) The above (1) whose peripheral velocity of the aforementioned cooling roller is 1-60m/second, or the manufacture method of a magnet material given in (2).

[0018] (4) The above (1) whose maximum eccentricity of the cooling roller peripheral surface accompanying rotation of the aforementioned cooling roller is below the double precision of the average thickness of a thin band-like magnet material obtained, or the manufacture method of a magnet material given in either of (3).

[0019] (5) The aforementioned controlled atmosphere is the manufacture method of a magnet material the above (1) which is inert gas, or given in either of (4).

[0020] (6) The aforementioned magnet material is the manufacture method of a magnet material the above (1) which is an alloy containing R (however, at least one sort in the rare earth elements in which R contains Y), or given in either of (5).

[0021] (7) The aforementioned magnet material is the manufacture method of a magnet material the above (1) which is an alloy containing R (however, at least one sort in the rare earth elements in which R contains Y), and TM (however, TM, at least one sort in transition metals) and B, or given in either of (5).

[0022] (8) Thin band-like magnet material characterized by being manufactured by the manufacture method of a magnet material the above (1) or given in either of (7).

[0023] (9) Powdered magnet material characterized by having ground the magnet material of a publication to the above (8), and supposing that it is powdered.

[0024] (10) The bond magnet which combines a powdered magnet material of a publication with the above (9) by the joint resin, and is characterized by the bird clapper.

[0025] (11) A bond magnet given in the above (10) whose content of the magnet material of the shape of aforementioned powder is 82 - 99.5wt%.

[0026] (12) Coercive force  $iH_c$  The above (10) which is 0.35 or more MA/m, or bond magnet given in (11).

[0027] (13) Magnetic-energy product (BH) max 50 kJ/m<sup>3</sup> The above (10) which it is above, or bond magnet given in either of (12).

[0028]

[Embodiments of the Invention] Hereafter, the manufacture method, the magnet material, and the bond magnet of the magnet material of this invention are explained in detail, referring to an accompanying drawing.

[0029] The perspective diagram showing the example of composition of the equipment (quenching thin band manufacturing installation) to which drawing 1 manufactures the magnet material of this invention by the single rolling method, and drawing 2 are the cross-section side elevations showing the state near the collision part to the cooling roller of the molten metal in the equipment shown in drawing 1.

[0030] As shown in drawing 1, the quenching thin band manufacturing installation 1 is equipped with the barrel 2 which can contain magnet material, and the cooling roller 5 which rotates in the direction of arrow in drawing A to this barrel 2. The nozzle (orifice) 3 which injects the molten metal of magnet material is formed in the soffit of a barrel 2.

[0031] Moreover, by arranging the coil 4 for heating at an about three nozzle [of a barrel 2] periphery, and impressing a RF to this coil 4, the inside of a barrel 2 is heated (IH) and the magnet material in a barrel 2 is changed into a melting state.

[0032] The cooling roller 5 consists of a base 51 and a surface layer 52 which forms the peripheral surface 53 of a cooling roller 5.

[0033] The component of a base 51 may consist of the quality of the materials which may really consist of the same quality of the materials as a surface layer 52, and are different in a surface layer 52.

[0034] Although especially the component of a base 51 is not limited, it is desirable to consist of metallic materials with high thermal conductivity like copper or a copper system alloy so that the heat of a surface layer 52 can be radiated more quickly.

[0035] Moreover, as for a surface layer 52, it is desirable to consist of metallic materials which are described below.

[0036] Such a quenching thin band manufacturing installation 1 is installed in a chamber (not shown), and operates in the state where it filled up with the controlled atmosphere of inert gas or others preferably in this chamber. In order to prevent oxidization of the quenching thin band 8 especially, as for a controlled atmosphere, it is desirable that it is inert gas.

[0037] As inert gas, although argon gas, gaseous helium, nitrogen gas, etc. are mentioned, for example, especially gaseous helium is desirable. the dimple according to the contamination of a gas stream to the roll side 81 of the quenching thin band 8 when gaseous helium is used for the reason as a controlled atmosphere, especially area -- the huge 2000-micrometer two or more dimples 13 (the imaginary line in drawing 2 shows) -- being generated -- being hard -- it is because heat transfer nature improves and higher magnetic properties are obtained

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[0038] When magnet ingredients are paid in a barrel 2, and it heats with a coil 4, it fuses in the quenching thin band manufacturing installation 1 and the molten metal 6 is injected from a nozzle 3, as shown in drawing 2, a molten metal 6 It is cooled quickly, and solidifies, being dragged by the peripheral surface 53 of the rotating cooling roller 5, after colliding with the peripheral surface 53 of a cooling roller 5 and forming a paddle (cold slug well) 7, and the quenching thin band 8 is formed continuously or intermittently. Thus, soon, the roll side 81 separates from a peripheral surface 53, and the formed quenching thin band 8 runs in the direction of arrow B in drawing 1. In addition, a dotted line shows the solidification interface 71 of a molten metal among drawing 2.

[0039] Although the suitable range changes with wettability to composition of an alloy molten metal, and the molten metal 6 of a peripheral surface 53 etc., usually, as for the peripheral velocity  $V$  of a cooling roller 5, it is desirable that it is 1-60m/second, and it is more desirable that it is 5-40m/second. If the peripheral velocity of a cooling roller 5 is too slow, thickness  $t$  of the quenching thin band 8 will become thick with the volumetric flow rate  $Q$  of the quenching thin band 8 (refer to the aforementioned formula (I)), the diameter of crystal grain will increase, if the peripheral velocity  $V$  of a cooling roller 5 is too quick conversely, it will become amorphous and, in any case, magnetic properties will fall.

[0040] The metallic material which constitutes the peripheral surface 53 of a cooling roller 5, i.e., the metallic material which constitutes a surface layer 52, (henceforth "roll peripheral surface material") has the wettability (only henceforth "wettability") to the following molten metals 6, and it is constituted. That is, as shown in drawing 3, a horizontal surface 10 is formed with the roll peripheral surface material 9, the drop of a molten metal 6 is placed on this horizontal surface 10, and when it is made to solidify, the contact angle  $\theta$  with the horizontal surface 10 of the congelation 11 to make becomes 70-170 degrees. In this case, it is desirable that it is 80-165 degrees, as for a contact angle  $\theta$ , it is more desirable that it is 90-160 degrees, and it is still more desirable that it is 95-150 degrees.

[0041] The wettability of the peripheral surface 53 of a cooling roller 5 is not measured directly, but a horizontal surface 10 is formed with the same material (roll peripheral surface material) as it here, and the wettability of this horizontal surface 10 is measured because it cannot stop and put the drop of a molten metal 6 on a fixed position but measurement of a contact angle is impossible or difficult for it, since a peripheral surface 53 is a curve convex.

[0042] In addition, in order to obtain more the correspondence relation with wettability of the peripheral surface 53 of the cooling roller 5 used for actual manufacture of a quenching thin band to accuracy on the occasion of measurement of a contact angle  $\theta$ , it is desirable to perform solidification of the drop of a molten metal 6 in the aforementioned controlled atmosphere, a homotypic, and the gas of this \*\*. Moreover, the volume of a congelation 11 is 3 0.005-0.1cm. Measuring in the range is desirable.

[0043] If a contact angle  $\theta$  exceeds the upper limit of the above-mentioned range, the wettability of a peripheral surface 53 will be bad, and it will become the inclination for thickness  $t$  of the quenching thin band 8 to become thick with the volumetric flow rate  $Q$  of the quenching thin band 8, especially the crystal grain by the side of the free side 82 of the quenching thin band 8 will turn big and rough, and magnetic properties will fall. In addition, if a volumetric flow rate  $Q$  is made small also by this case, although thickness  $t$  also becomes thin (refer to the aforementioned formula (I)) and this fault is canceled or eased, since the fall of productivity is caused, it is not desirable.

[0044] Moreover, since the wettability of a peripheral surface 53 is too good in a contact angle  $\theta$  being under the lower limit of the above-mentioned range, a paddle 7 spreads too much, therefore the configuration of the quenching thin band 8 and a size (width of face  $w$ , thickness  $t$ ) become unstable, and the uniform and homogeneous quenching thin band 8 is not obtained (variation arises in a state, magnetic properties, etc. of crystal grain).

[0045] In addition, on the occasion of measurement of a contact angle  $\theta$ , the ablation (coming floating) 12 as shown in drawing 4 may arise by the solidification shrinkage near the solidification interface the drop of a molten metal 6 contacts a horizontal surface 10. In this case, the portion which ablation 12 produced is excepted and a contact angle  $\theta$  is measured. Namely, a contact angle  $\theta$  is measured by using as datum level field 10' parallel to the horizontal surface 10 which passes along the upper limit (peak) of ablation 12.

[0046] By the way, in the quenching thin band manufacturing installation 1, a cooling roller 5 faces rotating, and as shown in drawing 5, some eccentricity (axial deflection) arises from the dimensional accuracy (roundness) of cooling roller 5 the very thing, the installation precision over the bearing of a cooling roller 5, etc.

[0047] If this eccentricity is large, the front face and the solidification interface 71 of a melting alloy in a paddle 7 vibrate, change will arise in the size (width of face  $w$ , thickness  $t$ ) of the obtained quenching thin band 8, or change will arise at the time when the roll side 81 of the quenching thin band 8 touches the peripheral surface 53 of a cooling roller 5. Furthermore, the incidence rate of the aforementioned huge dimple 13 also increases. Consequently, the cooling rate of the quenching thin band 8 etc. is changed, and variation arises in magnetic properties. And magnetic properties fall [ the bond magnet using the magnet powder or it which were obtained from such a quenching thin band 8 ].

[0048] In order to prevent such a thing, it is desirable to make maximum eccentricity  $\Delta R$  (to refer to drawing 5) of the peripheral surface 53 of the cooling roller 5 accompanying rotation of a cooling roller 5 below into the double precision of thickness (average)  $t$  of the quenching thin band 8 which can be obtained by this

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invention, considering as 1.5 or less times is more desirable, and considering as 1 or less time is still more desirable. Thereby, the magnetic properties of the obtained quenching thin band 8 can be made more into homogeneity. And the magnetic properties of the bond magnet manufactured from this can be raised. Especially in this invention, further excellent magnetic properties can be demonstrated according to the synergistic effect of specifying such maximum eccentricity  $\Delta R$  and specifying the wettability of the peripheral surface 53 mentioned above.

[0049] Here, especially the lower limit of maximum eccentricity  $\Delta R$  is the limitation of the precision of the bearing which supports the limitation of the process tolerance of the peripheral surface 53 of a cooling roller 5, and a cooling roller 5 although not limited to 0.1 micrometers. It can consider as a grade.

[0050] In addition, maximum eccentricity  $\Delta R$  can be measured with precision sizer vessels, such as for example, a laser displacement gage, an electrostatic displacement gage, and a precision gage.

[0051] As a magnet material in this invention, it is R (however, R). The alloy containing at least one sort in the rare earth elements containing Y, especially R (however, R) Rare earth permanent magnet material like the alloy containing at least one sort in the rare earth elements containing Y, and TM (however, TM, at least one sort in transition metals) and B is mentioned, and the thing of composition of following [1] - [4] is desirable.

[0052] [1] What makes a fundamental component the rare earth elements which are mainly concerned with Sm, and the transition metals which are mainly concerned with Co (henceforth a Sm-Co system alloy).

[0053] [2] What makes a fundamental component the transition metals which are mainly concerned with R (however, at least one sort in the rare earth elements in which R contains Y), and Fe, and B (henceforth a R-Fe-B system alloy).

[0054] [3] What makes a fundamental component the rare earth elements which are mainly concerned with Sm, the transition metals which are mainly concerned with Fe, and the element between grids which is mainly concerned with N (henceforth an Sm-Fe-N system alloy).

[4] What makes a fundamental component transition metals, such as R (at least one sort however, among the rare earth elements in which R contains Y), and Fe, and has a magnetic phase on nano meter level (nano crystal magnet).

[0055] As a typical thing of a Sm-Co system alloy, SmCo<sub>5</sub> and Sm<sub>2</sub>TM<sub>17</sub> (however, TM, transition metals) are mentioned.

[0056] As a typical thing of a R-Fe-B system alloy, a Nd-Fe-B system alloy, a Pr-Fe-B system alloy, a Nd-Pr-Fe-B system alloy, a Ce-Nd-Fe-B system alloy, a Ce-Pr-Nd-Fe-B system alloy, the thing that replaced a part of Fe in these by other transition metals, such as Co and nickel, are mentioned.

[0057] It is Sm<sub>2</sub>Fe<sub>17</sub>N<sub>3</sub> which nitrided and produced Sm<sub>2</sub>Fe<sub>17</sub> alloy as a typical thing of an Sm-Fe-N system alloy. It is mentioned.

[0058] as the aforementioned rare earth elements, Y, La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, and a misch metal mention -- having -- these -- one sort -- or two or more sorts can be included moreover, Fe, Co, nickel, etc. mention as the aforementioned transition metals -- having -- these -- one sort -- or two or more sorts can be included Moreover, in order to raise magnetic properties, in magnet material, B, aluminum, Cu, Ga, Si, Ti, V, Ta, Zr, Nb, Mo, Hf, Ag, Zn, P, germanium, etc. can also be contained if needed.

[0059] The magnetic properties in which the quenching thin band (thin band-like magnet material) 8 of this invention obtained by the above manufacture methods turned minutely, consequently crystal grain was excellent are obtained.

[0060] Moreover, a powdered magnet material (magnet powder) of this invention is obtained by grinding such a quenching thin band 8.

[0061] Especially the method of trituration is not limited, for example, can be performed using various trituration equipments, such as a ball mill, a vibration mill, a jet mill, and a pin mill, and a shredding equipment. In this case, trituration can also be performed in a non-oxidizing atmosphere like [ under a vacuum or a reduced pressure state (for example,  $1 \times 10^{-1}$  -  $1 \times 10^{-6}$  Torr) or in inert gas, such as nitrogen gas argon gas, and gaseous helium, ], in order to prevent oxidization.

[0062] A different thing only not only in the thing of the same composition which mixed the magnet powder of two or more sorts of composition is sufficient as such magnet powder. For example, what mixed at least two sorts in the thing of composition of aforementioned [1] - [4] is mentioned. In this case, it can have the advantage of each magnet powder to mix simulataneously, and more excellent magnetic properties can be obtained easily.

[0063] Moreover, in the case of the thing for manufacturing the bond magnet mentioned later, although not limited, especially the mean particle diameter of magnet powder is 0.5-60 micrometers. A grade is desirable and it is 1-40 micrometers. A grade is more desirable. moreover, in order to obtain the good moldability at the time of fabrication by a small amount of joint resin which is mentioned later, the particle size distribution of magnet powder is distributed to some extent -- \*\*\*\*'s (there is variation) -- it is desirable The rate of a hole of the obtained bond magnet can be reduced by this, the mechanical strength of a bond magnet can be raised more, and magnetic properties can be improved further.

[0064] In addition, the mean particle diameters may differ for every composition of different magnet powder case [ powder ] and mixed although the magnet powder of two or more sorts of composition was mixed. Moreover, what is necessary is to just be manufactured by the method of a different this invention which at least one sort in the magnet powder of two or more sorts of composition mentioned above the case in the end of

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such mixed powder.

[0065] When a bond magnet is manufactured using the above magnet powder, such magnet powder has good unity (wettability of a joint resin) with a joint resin, therefore this bond magnet has a high mechanical strength and it becomes the thing excellent in thermal stability (thermal resistance) and corrosion resistance. Therefore, the magnet powder concerned fits manufacture of a bond magnet.

[0066] In addition, the magnet powder (powdered magnet material) of this invention cannot be overemphasized by that you may be what it is not limited to what is used for manufacture of a bond magnet, for example, is used for manufacture of a sintered magnet.

[0067] Next, the bond magnet of this invention is explained.

[0068] The bond magnet of this invention comes to join the above-mentioned magnet powder together by the joint resin.

[0069] As a joint resin (binder), any of thermoplastics and thermosetting resin are sufficient.

[0070] As thermoplastics, for example A polyamide (example : nylon 6, Nylon 46, Nylon 66, Nylon 610, Nylon 612, Nylon 11, Nylon 12, Nylon 612, nylon 6-66), Liquid crystal polymers, such as a thermoplastic polyimide and an aromatic polyester, a polyphenylene oxide, Polyolefines, such as polyphenylene sulfide, polyethylene, polypropylene, and an ethylene vinylacetate copolymer, A denaturation polyolefine, a polycarbonate, a polymethylmethacrylate, Polyester, such as a polyethylene terephthalate and a polybutylene terephthalate, The copolymer which is mainly concerned with these, a blend object, a polymer alloy, etc. are mentioned, and a polyether, a polyether ether ketone, polyether imide, a polyacetal, etc. can mix and use 1 of sorts of these, and two sorts or more.

[0071] Also among these, especially the moldability is excellent, and since the mechanical strength is high, that which is mainly concerned with a liquid crystal polymer and polyphenylene sulfide from the point of a polyamide and heat-resistant improvement is desirable. Moreover, these thermoplastics is excellent also in kneading nature with magnet powder.

[0072] Such thermoplastics has the advantage that selection wide range like what thought the moldability as important, and the thing which thought thermal resistance and the mechanical strength as important is attained by the kind, copolymerization-ization, etc.

[0073] On the other hand, as thermosetting resin, various epoxy resins, such as a bisphenol type, a novolak type and a naphthalene system, phenol resin, a urea resin, melamine resin, a polyester (unsaturated polyester) resin polyimide resin, silicone resin, a polyurethane resin, etc. are mentioned, and 1 of sorts of these and two sorts or more can be mixed and used, for example.

[0074] From the point of especially the moldability being excellent also among these, and a mechanical strength being high and excelling in thermal resistance, an epoxy resin, phenol resin, polyimide resin, and silicone resin are desirable, and especially an epoxy resin is desirable. Moreover, these thermosetting resin is excellent also in kneading nature with magnet powder, and the homogeneity of kneading.

[0075] In addition, the thing of a solid [ thing / liquefied ] (powdered) is sufficient as the thermosetting resin (un-hardening) used at a room temperature.

[0076] Such a bond magnet of this invention is manufactured as follows, for example. Magnet powder, a joint resin, and the constituent for bond magnets (compound) that contains additives (an antioxidant, lubricant, etc.) if needed are manufactured, and it fabricates in a desired magnet configuration all over a magnetic field or a non-magnetic field by methods, such as compression molding, extrusion molding, and injection molding, using this constituent for bond magnets. When a joint resin is thermosetting resin, it is hardened by heating etc. after fabrication.

[0077] As for the content of the magnet powder in a bond magnet, it is desirable that it is about 82-99.5wt%, and it is more desirable that it is about 90-99wt%. Although the bond magnet was especially manufactured by compression molding, as for the content of magnet powder, to a case, it is desirable that it is about 93-99.5wt%, and it is more desirable to it that it is about 95-99wt%.

[0078] If improvement in magnetic properties (especially magnetic-energy product) cannot be aimed at if there are too few contents of magnet powder, and there are too many contents of magnet powder, joint resins content will decrease relatively and a moldability will fall.

[0079] Such a bond magnet of this invention demonstrates outstanding magnetic properties from the property of the quenching thin band 8 used as the raw material mentioned above, the numerousness of the manufacture conditions of a bond magnet, and the contents of the magnet powder contained in a bond magnet, etc.

[0080] That is, the bond magnet of this invention is coercive force  $iH_c$ . 0.35 or more MA/m is 0.50 or more MA/m more preferably.

[0081] The bond magnet of this invention, especially the bond magnet fabricated all over the non-magnetic field are magnetic-energy (product BH) max. It is 50 kJ/m<sup>3</sup> preferably. It is 70 kJ/m<sup>3</sup> more preferably above. It is above.

[0082] It is not limited, for example, about a configuration, the thing of all configurations, such as the shape of the shape of a pillar, a prismatic, and a cylinder (the shape of a ring), circular, plate-like, and a curve tabular, is possible for the configuration of the bond magnet of this invention, especially a size, etc., for example, and the thing of all sizes is possible for them from a thing also with the large-sized size to a micro thing.

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## EXAMPLE

[Example] Hereafter, the concrete example of this invention is explained.

[0084] (Example 1) The hardener ingot as which alloy composition is expressed in Nd<sub>10</sub>Pr<sub>2.5</sub> Fe<sub>bal</sub>.Co<sub>6</sub> aluminum<sub>3</sub> Cu<sub>1.5</sub> Nb<sub>1</sub> Ga<sub>1</sub> B<sub>5</sub> (composition A) was cast. About 15g sample was started from this ingot.

[0085] The quenching thin band manufacturing installation 1 of composition of being shown in drawing 1 was prepared, and the aforementioned sample was put in in the quartz tube which prepared the nozzle (circular hole orifice) in the pars basilaris ossis occipitalis. After deaerating the inside of the chamber by which the quenching thin band manufacturing installation 1 is contained, gaseous helium was introduced as a controlled atmosphere and it considered as the temperature of 21 degrees C, and the controlled atmosphere of pressure 60KPa.

[0086] Then, the ingot sample in a quartz tube was fused by high-frequency induction heating, this molten metal was injected by the differential pressure of the internal pressure of a quartz tube, and an ambient pressure towards the peripheral surface of the cooling roller with a diameter [ of 200mm ], and a width of face of 20mm which rotates by 1500rpm (peripheral velocity : 15.7m/(second)), and the quenching thin band of the alloy of the aforementioned composition A was obtained.

[0087] The surface layer (roll peripheral surface) of a cooling roller should consist of Pd-8wt%Ru-2wt%Pt alloys. Moreover, thickness of this surface layer was set to 5mm.

[0088] It was 95 degrees when the contact angle theta of the aforementioned congelation was measured by the method which forms a horizontal surface with the component and this material of this surface layer, trickles the molten metal of the aforementioned composition A calmly, is made to solidify it on the aforementioned horizontal surface in the gas of the aforementioned controlled atmosphere and these conditions, obtains a congelation (volume 0.01cm<sup>3</sup>), and is shown in drawing 3 or drawing 4 . In addition, measurement of a contact angle theta was optically performed using the projector.

[0089] moreover, the place which measured maximum eccentricity deltaR of the cooling roller peripheral surface by rotation of a cooling roller by the laser displacement gage -- deltaR=10micrometer it was .

[0090] (Example 2) The quenching thin band was manufactured like the example 1 except having constituted the surface layer (roll peripheral surface) of a cooling roller from a nickel-10wt%Ti-10wt%aluminum-5wt%Mo alloy.

[0091] It was 150 degrees, when the horizontal surface was formed with the component and this material of this surface layer, the molten metal of the aforementioned composition A is dropped calmly, was made to solidify on the aforementioned horizontal surface in the gas of the aforementioned controlled atmosphere and these conditions, the congelation (volume 0.01cm<sup>3</sup>) was obtained and the contact angle theta of the aforementioned congelation was measured by the same method as an example 1.

[0092] moreover, the place which measured maximum eccentricity deltaR of the cooling roller peripheral surface by rotation of a cooling roller by the laser displacement gage -- deltaR=12micrometer it was .

[0093] (Example 3) While manufacturing a quenching thin band from the molten metal of this composition which used the ingot which consists of Nd<sub>11</sub>Ce<sub>2</sub> Sm<sub>1</sub> Fe<sub>bal</sub>.Co<sub>4</sub> Cu<sub>1.5</sub> Ga<sub>1</sub> Ti<sub>0.5</sub> B<sub>6</sub> (composition B) The quenching thin band was manufactured like the example 1 except having constituted the surface layer (roll peripheral surface) of a cooling roller from a W-20wt%Zr-3wt%Nb alloy.

[0094] It was 70 degrees, when the horizontal surface was formed with the component and this material of this surface layer, the molten metal of the aforementioned composition B is dropped calmly, was made to solidify on the aforementioned horizontal surface in the gas of the aforementioned controlled atmosphere and these conditions, the congelation (volume 0.01cm<sup>3</sup>) was obtained and the contact angle theta of the aforementioned congelation was measured by the same method as an example 1.

[0095] moreover, the place which measured maximum eccentricity deltaR of the cooling roller peripheral surface by rotation of a cooling roller by the laser displacement gage -- deltaR=9micrometer it was .

[0096] About each quenching thin band of the <characterization of quenching thin band> examples 1-3, the width of face w and thickness t were measured. This measurement was measured by 20 point of measurement per quenching thin band by the microscope, and was made into the value which averaged this, respectively.

[0097] Next, while measuring the diameter of average crystal grain from the organization observation result by transverse electromagnetic about each quenching thin band, magnetic properties (coercive force iHc and magnetic-energy (product BH) max) were measured by VSM.

[0098] These measurement results are shown in the following table 1.

[0099] In addition, there was very little variation by the measurement part (less than \*\*5% of averages), and each size (width of face w, thickness t) of each quenching thin band had high dimensional stability.

[0100] Moreover, when the rate of area which observes a roll side with a scanning electron microscope (SEM), and performs image analysis further about each quenching thin band, and the with a 2000-micrometer area [ or more 2 ] huge dimple to a roll side occupies from this analysis result was investigated, all were low values very much.

[0101]

[Table 1]

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## 急冷薄帯の特性

	接 触 角 $\theta$	急冷薄帯の幅 $w$ (mm)	急冷薄帯の厚さ $t$ ( $\mu\text{m}$ )	平均結晶粒径 (nm)	$iH_c$ (kA/m)	(BH) max (kJ/m <sup>3</sup> )
実施例 1	95°	1.1	26.7	25	0.69	112
実施例 2	150°	0.8	30.9	29	0.64	104
実施例 3	70°	1.4	24.5	23	0.95	92

[0102] As shown in Table 1, each quenching thin band of this invention of examples 1-3 can attain detailed-ization of crystal grain, and high magnetic properties are obtained.

[0103] (Example 4) A grinder (RAIKAI machine) grinds the quenching thin band of an example 1 in inert gas, and a mean particle diameter is 16 micrometers. It considered as magnet powder, this magnet powder, epoxy resin 2.0wt%, hydrazine system antioxidant 0.15wt%, and stearate (lubricant) 0.05wt% were mixed, this mixture was fully kneaded (120 degree-Cx 10 minutes), and the constituent for bond magnets (compound) was produced.

[0104] subsequently -- grinding this compound and being granular -- carrying out -- this granular object -- weighing capacity -- carrying out -- the metal mold of press equipment -- inside -- being filled up -- 130 degrees C of material temperature, and pressure 6 ton/cm<sup>2</sup> It pressed (inside of a non-magnetic field), and the Plastic solid was obtained. Heat hardening of the epoxy resin was carried out after mold release, and the pillar-like bond magnet with a diameter [ of 10mm ] x height of 7mm was obtained.

[0105] (Example 5) A grinder (RAIKAI machine) grinds the quenching thin band of an example 2 in inert gas, and a mean particle diameter is 20 micrometers. It considered as magnet powder, this magnet powder, epoxy resin 2.5wt%, hydrazine system antioxidant 0.1wt%, and stearate (lubricant) 0.1wt% were mixed, this mixture was fully kneaded (120 degree-Cx 10 minutes), and the constituent for bond magnets (compound) was produced

[0106] subsequently -- grinding this compound and being granular -- carrying out -- this granular object -- weighing capacity -- carrying out -- the metal mold of press equipment -- inside -- being filled up -- 130 degrees C of material temperature, and pressure 6 ton/cm<sup>2</sup> It pressed (inside of a non-magnetic field), and the Plastic solid was obtained. Heat hardening of the epoxy resin was carried out after mold release, and the pillar-like bond magnet with a diameter [ of 10mm ] x height of 7mm was obtained.

[0107] (Example 6) A grinder (RAIKAI machine) grinds the quenching thin band of an example 3 in inert gas, and a mean particle diameter is 18 micrometers. It considered as magnet powder, this magnet powder, epoxy resin 1.9wt%, hydrazine system antioxidant 0.1wt%, and stearate (lubricant) 0.05wt% were mixed, this mixture was fully kneaded (120 degree-Cx 10 minutes), and the constituent for bond magnets (compound) was produced.

[0108] subsequently -- grinding this compound and being granular -- carrying out -- this granular object -- weighing capacity -- carrying out -- the metal mold of press equipment -- inside -- being filled up -- 130 degrees C of material temperature, and pressure 6 ton/cm<sup>2</sup> It pressed (inside of a non-magnetic field), and the Plastic solid was obtained. Heat hardening of the epoxy resin was carried out after mold release, and the pillar-like bond magnet with a diameter [ of 10mm ] x height of 7mm was obtained.

[0109] (Example 7) The magnet powder obtained in the example 4 and the magnet powder obtained in the example 6 were uniformly mixed by the weight ratio 6:4, and mixed magnet powder was obtained. This mixed magnet powder, epoxy resin 2.0wt%, hydrazine system antioxidant 0.15wt%, and stearate (lubricant) 0.05wt% were mixed, this mixture was fully kneaded (120 degree-Cx 10 minutes), and the constituent for bond magnets (compound) was produced.

[0110] subsequently -- grinding this compound and being granular -- carrying out -- this granular object -- weighing capacity -- carrying out -- the metal mold of press equipment -- inside -- being filled up -- 130 degrees C of material temperature, and pressure 6 ton/cm<sup>2</sup> It pressed (inside of a non-magnetic field), and the Plastic solid was obtained. Heat hardening of the epoxy resin was carried out after mold release, and the pillar-like bond magnet with a diameter [ of 10mm ] x height of 7mm was obtained.

[0111] (Example 8) The magnet powder obtained in the example 4, the magnet powder obtained in the example 5, and the magnet powder obtained in the example 6 were uniformly mixed by the weight ratio 2:3:5, and mixed magnet powder was obtained. This mixed magnet powder, epoxy resin 1.8wt%, hydrazine system antioxidant 0.2wt%, and stearin acid (lubricant) 0.1wt% were mixed, this mixture was fully kneaded (120 degree-Cx 10 minutes), and the constituent for bond magnets (compound) was produced.

[0112] subsequently -- grinding this compound and being granular -- carrying out -- this granular object --

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weighing capacity -- carrying out -- the metal mold of press equipment -- inside -- being filled up -- 130 degrees C of material temperature, and pressure 6 ton/cm<sup>2</sup> It pressed (inside of a non-magnetic field), and the Plastic solid was obtained. Heat hardening of the epoxy resin was carried out after mold release, and the pillar-like bond magnet with a diameter [ of 10mm ] x height of 7mm was obtained.

[0113] About each bond magnet of the <characterization of bond magnet> examples 4-8, the magnetic properties (coercive force iH<sub>c</sub> and magnetic-energy (product BH) max) were measured in maximum impression magnetic field 2 MA/m with the account fluxmeter of \*\*\*\*\*.

[0114] furthermore -- these bond magnets -- 60 degree-Cx95%RH -- the constant temperature by 500 hours -- the constant humidity examination was performed and corrosion resistance was investigated This corrosion resistance distinguished the existence of generating of the rust in a bond magnet front face by viewing, and evaluated as a x mark what \*\* mark and generating of rust were notably accepted in in what O mark and generating of rust were accepted in a little in the thing without generating of rust.

[0115] These measurement results are shown in the following table 2. Moreover, the content (in the case of mixed magnet powder, it is the total amount) of the magnet powder in each bond magnet is collectively described all over the following table 2.

[0116]

[Table 2]

ボンド磁石の特性

	用いた磁石粉末	ボンド磁石中の 磁石粉末含有量 (wt%)	i H <sub>c</sub> (MA/m)	(BH) max (kJ/m <sup>3</sup> )	耐食性
実施例 4	実施例 1	97.9	0.67	73.5	○
実施例 5	実施例 2	97.4	0.62	70.6	△~○
実施例 6	実施例 3	98.0	0.94	62.9	○
実施例 7	実施例 1、3の混合 (=6:4)	97.9	0.84	70.4	○
実施例 8	実施例 1、2、3の混合 (=2:3:5)	98.1	0.90	68.5	○

[0117] As shown in Table 2, each bond magnet of this invention of examples 4-8 is more than coercive force iH<sub>c</sub> 0.35 MA/m and magnetic-energy (product BH) max. 50 kJ/m<sup>3</sup> Corrosion resistance is also excellent while having the magnetic properties which were excellent with the above.

[0118] Especially, more excellent magnetic properties are obtained in the examples 7 and 8 using mixed magnet powder.

## DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] It is the perspective diagram showing the example of composition of the equipment (quenching thin band manufacturing installation) which manufactures the magnet material of this invention.

[Drawing 2] It is the cross-section side elevation showing the state near the collision part to the cooling roller of the molten metal in the equipment shown in drawing 1.

[Drawing 3] It is the cross-section side elevation showing the wettability measuring method for the molten metal of a cooling roller peripheral surface.

[Drawing 4] It is the cross-section side elevation showing the wettability measuring method for the molten metal of a cooling roller peripheral surface.

[Drawing 5] It is the side elevation showing the maximum eccentricity of the cooling roller peripheral surface accompanying rotation of a cooling roller.

[Description of Notations]

1 Quenching Thin Band Manufacturing Installation

2 Barrel

3 Nozzle

4 Coil

5 Cooling Roller

51 Base

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52 Surface Layer  
53 Peripheral Surface  
6 Molten Metal  
7 Paddle  
71 Solidification Interface  
8 Quenching Thin Band  
81 Roll Side  
82 Free Side  
9 Roll Peripheral Surface Material  
10 Horizontal Surface  
10' Field  
11 Congelation  
12 Ablation  
13 Huge Dimple

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